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IN ALL ITS APPLICATIONS TO

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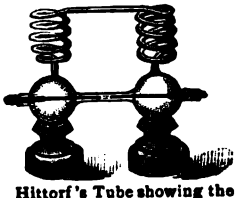

Hittorf's Tube showing the resistance of the dark space.

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No. 2197.—JANUARY 3, 1902.

CONTRIBUTIONS TO THE CHEMISTRY OF CHLOROPHYLL.

No. VIII. Changes Undergone by Chlorophyll in Passing Through the Bodies of Animals.*

By EDWARD SCHUNCK, F.R.S.

Considering the large quantity of food containing chlorophyll consumed by animals feeding on green herbage, it seemed to me it might be of interest to ascertain whether any, and if so what, changes are undergone by the chlorophyll of the food during its passage through the animal body.

It is stated in works on physiological chemistry that the solid excrements of animals contain chlorophyll; but this statement refers apparently to human fæces, and is certainly not correct as regards those of herbivora if unchanged chlorophyll is meant. It would indeed, *à priori*, seem very improbable that chlorophyll, after exposure at a somewhat elevated temperature to acids and other agents, such as it would meet with in its passage through the animal system, would remain unchanged, though, on the other hand, chlorophyll products of decomposition, some of which, as I have pointed out on former occasions, are very stable bodies, might be looked for in the excrements of animals.

Having treated some of the fæces of a cow that had been fed for some time on grass only, with boiling alcohol, I obtained a dark greenish-brown extract showing an acid reaction, a quantity of undigested matter consisting of stems, woody fibre, &c., being left undissolved. A little of the filtrate, on being mixed with water and shaken up with ether, gave a golden-yellow supernatant liquid, which, if chlorophyll had been present in the material used, would have shown a decided green colour, and would have exhibited the absorption spectrum of chlorophyll. In place of the latter, however, it showed the four absorption bands of phylloxanthin, as well marked, indeed, as I have ever seen them. This simple experiment proved conclusively the absence of chlorophyll, but in its place the presence of one of its products of decomposition. I have made no attempt to isolate and purify this supposed phylloxanthin, this being rendered difficult owing to the large admixture of impurities, which are chiefly of a fatty nature. The ethereal liquid just referred to would also have contained the urobilin of the fæces if present, so I imagine at least if I have rightly understood what has been stated regarding that body. The properties of urobilin, however, are not very marked. It is only by its absorption spectrum that it can with certainty be detected, but this spectrum is too inconspicuous and too faint to be easily seen in a solution containing at the same time phylloxanthin with its dark well-defined bands, which would completely mask those of urobilin situated as these are in the same region of the spectrum. Its presence, too, if proved, would have been of little interest from my point of view, and I therefore made no attempt to establish its presence or absence.

The extract of fæces with boiling alcohol gave, after filtration and cooling, a dark coloured flocculent deposit, which was filtered off, dried, and treated with boiling chloroform. The filtered chloroformic liquid left on evaporation a quantity of purplish-blue lustrous crystals. These crystals, which are of considerable interest as regards both their properties and their origin, will be described presently. They are more readily prepared by extracting cow-dung after pressure between folds of paper with cold chloroform, filtering, and evaporating the filtrate slowly in a warm place, when the substance separates in the form of brilliant semi-metallic spangles floating in the liquid, which, after collecting and washing with alcohol, have the appearance of a lustrous crystalline mass. From the brilliancy of its appearance an observer might easily be deceived as regards its quantity, which is not really large. I have, however, obtained sufficient to enable me to determine its chief properties, and to justify the conclusion that it is a derivative of chlorophyll closely resembling, though not identical with, phyllocyanin, as I shall presently show.

For such as wish to prepare this substance, I may state, as the result of my experience, that it can only be obtained from the fæces of cows or other herbivora that have been fed exclusively on grass or other green vegetable food; those of stall-fed cattle, nourished as they usually are to a great extent with oilcake, yield none, on account perhaps of the large amount of fatty matter present in that case in the fæces. I may add that the solid excrements of sheep that have been at pasture for some time and living on herbage only, on being treated as above described, yield the same substance, but I have made no experiments with the fæces of other herbivora. I imagine that the yield of this peculiar substance is greatest from material collected in spring or early summer, but this, if correct, may be due to other causes than difference of season.

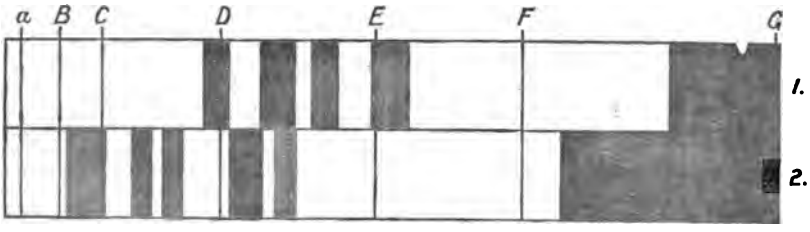
This substance not having, so far as I can ascertain, been previously observed, I propose to call *Scatocyanin*, a name kindly suggested to me by Professor Wilkins, of Owens College. Its chief properties are as follows:—Under the microscope it appears in the form of thin rhombic plates or elongated flat prismatic crystals, which are pale brown by transmitted light, of a purplish-blue colour with a brilliant metallic lustre by reflected light. When heated between watch-glasses it is decomposed without melting or swelling much or yielding any subli-

* A Paper read before the Royal Society, December 12, 1901.

mate; heated further on platinum, it burns away, leaving a little ash. It is almost insoluble in boiling alcohol, ether, carbon disulphide, and benzol, but it dissolves, though not readily, in chloroform, giving a solution which shows an absorption spectrum of five bands, almost identical with those of phyllocyanin. It dissolves in boiling glacial acetic acid, giving a fine crimson solution, which, when sufficiently dilute, shows an absorption spectrum of four bands—of which the two first are well defined, the third faint with some obscuration between it and the second band, the fourth band also faint and not well defined (see Fig.) From a saturated solution in boiling acetic acid the substance separates on cooling and standing in lustrous purplish-blue needles.

It dissolves in concentrated sulphuric acid with a brilliant grass-green colour, which, on standing, changes to purplish-blue. The solution now shows a characteristic absorption spectrum of five bands, of which the first and fifth are faint and poorly defined, the second and third well defined, but the fourth only moderately so, while there is much obscuration between the fourth and fifth bands, with just a trace of a sixth band in the green between the fifth band and the total obscuration (see Fig.)

The sulphuric acid solution on being mixed with several times its volume of water changes its colour from purplish-blue to a fine purple without giving any precipitate, and



ABSORPTION-SPECTRA OF SCATOCYANIN SOLUTIONS.

1. Scatocyanin in glacial acetic acid. 2. The same in concentrated sulphuric acid. (These spectra were mapped by my son, Charles A. Schunck).

without showing any change in its absorption spectrum. On standing, however, for some time the liquid becomes nearly colourless, and deposits rosettes of lustrous crystalline needles, presumably of unchanged substance; so that it appears no sulphonic acid is formed in this reaction, the colour of the solution in sulphuric acid being due probably to a loose combination of the substance with the acid. The substance is soluble in boiling aniline; the solution is dull red, and gives with alcohol a deposit of brilliant needle-shaped crystals, the filtrate from which shows the same spectrum as the solution in acetic acid. The substance is insoluble in aqueous caustic potash, but dissolves in alcoholic potash, giving a yellow solution.

There is another substance of definite character, and assuming a crystalline form which I have obtained, though not always, along with the preceding. Having treated the fæces of a cow that had lived for some time on green vegetable food, with acidulated alcohol (seventeen parts of rectified spirit to three of sulphuric acid, the mixture prescribed for the preparation of so-called stercobilin), I added water to the filtered extract, and shook up with chloroform. The chloroformic liquid after separation was evaporated, when it left a brown syrup. This, treated with boiling alcohol, dissolved in part, a semi-crystalline pink residue being left undissolved, which, after separation, was found to be soluble in boiling acetic acid with a crimson colour, and consisted doubtless of scatocyanin. A further quantity of this was deposited on again evaporating; on further evaporation, the filtrate left a thin brown syrup which was mixed with a large quantity of alcohol. On standing, a voluminous crystalline deposit separated, which was filtered off, and slightly washed with alcohol.

The following are the chief properties of the substance so prepared:—When dry it has the appearance of a dull red mass of crystalline needles. Heated on platinum, it melts easily to a brown mass, solidifying again on cooling; on further heating, it gives off red fumes, and burns away, leaving no ash. When heated in a test-tube or between watch-glasses, it gives a small quantity of amorphous brown sublimate with a few crystalline needles. It dissolves in boiling alcohol, giving a bright yellowish-red solution, which, on cooling, deposits crystalline needles, becoming almost colourless. It is moderately soluble in ether, benzol, and glacial acetic acid; very soluble in chloroform. The chloroformic solution shows no absorption bands, only obscuration in the blue. It is quite insoluble in caustic potash liquor, even on boiling. It dissolves in concentrated sulphuric acid, giving a dull red solution, which, after the addition of a considerable quantity of acid, shows no absorption bands, only obscuration in the blue and green; on the addition of water, the solution turns reddish-yellow without giving any precipitate, but on standing for a short time the colour turns to a fine violet, and then shows a broad ill-defined absorption band between the red and green. Not showing any very characteristic reaction but the one last mentioned, it must remain doubtful how and where it originates. It may possibly be a constituent of the green parts of plants not hitherto observed, though I believe it myself to be a derivative of chlorophyll—meaning by chlorophyll the *ensemble* of the colouring matters of green leaves—formed by some unknown process in the animal economy, but the fact of its solutions showing no absorption bands does not lend countenance to this view. Having also observed it on one occasion only, I do not feel justified in giving it a name, or in placing it in any known category of vegetable or animal colouring matters.

Another constituent of the fæces remains to be mentioned. It was referred to above as showing in solution the absorption spectrum of phylloxanthin. After all the substances capable of assuming a crystalline form have been separated from the alcoholic extract of the fæces, this phylloxanthin-like substance is found in the final mother-liquor. I have not succeeded in obtaining it in a crystalline or any other definite form, on account probably of the large quantity of fatty matter with which it is associated; but there is no reason, I think, to suppose that it differs essentially from the phylloxanthin described in previous communications as a product of the action of acids on chlorophyll.

The conclusions to which the experiments above described lead may be summarised as follows:—

1. The fæces of animals supplied with green vegetable food only—such at least as have so far been examined—contain no chlorophyll, but in its place substances which must be supposed to be derivatives of chlorophyll, formed partly by the action of acids on the chlorophyll of the food, partly by some agency to which the latter is subjected in its passage through the body.

2. Of these substances, one seems to be identical with phylloxanthin, a well known product of decomposition of chlorophyll. Another is a substance of well-marked properties, nearly resembling, but not identical with,

phyllocyanin. It has not, so far as my experience goes, been hitherto observed as a result of any process of decomposition to which chlorophyll has been subjected outside the animal body. I consider it as a body *sui generis*, characterised by its fine purplish-blue colour and its brilliant metallic lustre. The existence of other products in addition to these two is possible. On one occasion, indeed, a definite crystalline substance was obtained, which seemed to be peculiar, but that it was in any way connected with chlorophyll could not with certainty be maintained.

NOTE ON THE SESQUITERPENE OF EUCALYPTUS OILS.*

By HENRY G. SMITH, F.C.S., Assistant Curator, Technological Museum.

In this paper the author showed that a sesquiterpene occurs in many Eucalyptus oils, and that it is this constituent that gives the pink colouration to Eucalyptus oil when testing for eucalyptol with phosphoric acid. In the oil of *E. hæmastoma* the sesquiterpene occurs in large amount, over 50 per cent of the crude oil distilling above 255° C. It is also present in quantity in the oils of several other species. Crystallised chemical products could not be obtained with it by the methods used. It is characterised by a range of five colour reactions which it gives with acids and with bromine when dissolved in glacial acetic acid. When the vapour of bromine is allowed to fall into the tube containing such a mixture, immediately it touches the liquid a crimson colour is formed, quickly changing to violet, and finally to a deep indigo-blue. It boils under atmospheric pressure at 260—265° C., has a specific gravity 0·9249 at 19° C., as obtained by repeated fractional distillation finally over sodium. Combustion results gave the terpene formula, and a vapour density determination showed it to be a sesquiterpene. The name *Aromadendrene* has been proposed for it.

BISMUTH ASSAY.

By A. W. WARWICK and T. D. KYLE.

Owing to the high price paid for bismuth, persistent search is being made for that metal; the result is that the localities in which it is known are rapidly increasing, especially in Colorado. In Leadville alone it is safe to say that nearly every mine has some bismuth in it, although few mines have ore rich enough to ship as bismuth. Most of the assay manuals give but scant attention to the bismuth assay, and the methods given are either inaccurate, or clumsy and laborious. For example, Furman's Manual gives a combined fire and gravimetric method which he admits to be inaccurate, but claims to be accurate enough for all technical purposes. We find that this method is not correct to within 0·75 per cent and that error amounts to 11·25 dol. per ton on a 10 per cent ore—an error too great for any good commercial work. It is also very laborious, taking from two to three hours to perform, which is a very serious objection in an ordinary assay office. Beringer's text-book gives the usual gravimetric method as well as a fire assay, remarking that the volumetric methods offer no advantages over the gravimetric method.

The fire assay is notoriously inaccurate, and the gravimetric method usually followed is too slow; we believe, therefore, that an account of a simple volumetric assay which is accurate will be of interest. It may also help to prevent those annoying disputes between buyer and seller which occur owing to differences in assay methods.

The price paid for bismuth is quite high, varying from 15 dol. to 20 dol. per unit per ton according to market price for bismuth, grade and quality of ore; an error, therefore, of 0·1 per cent amounts to from 1·50 dol. to 2 dol. per ton, showing that any bismuth assay should be very accurate. A good method of assay for bismuth is a desideratum, and it should not only be accurate, but simple and rapid, and also, if of the volumetric order, the standard solution used should be reasonably permanent. The writers have found these conditions fulfilled by a modification of the process devised by Pattison Muir some years since. It is rapid, 40 minutes sufficing to complete a determination. It is accurate, results agreeing to within 0·1 per cent with ordinary care; with extreme care results agree to within 0·05 per cent. It is simple; no delicate manipulations are required. The standard solution is the inevitable potassium permanganate; the usual standard solution used in the iron determinations can be used (1 c.c. = 0·010 grm. Fe), although for very accurate work a dilute solution is better. A permanganate solution in which 1 c.c. = 0·010 grm. Fe, will be equal to 0·0186 grm. bismuth; by diluting 100 c.c. of this with 86 c.c. of water a solution of permanganate will be obtained of which 1 c.c. should equal 0·010 grm. of bismuth. A permanganate solution 1 c.c. = 0·010 grm. Fe; found = 0·01868 grm. Bi. 100 c.c. permanganate above + 86 c.c. of water; 1 c.c. found = 0·01017 grm. Bi.

The process consists of the following operations:—(1) Solution in nitric acid, (2) precipitation as bismuth-oxalate, (3) conversion of the normal oxalate into basic-oxalate by boiling with water, (4) solution of the basic bismuth oxalate, (5) titrating the hot solution with potassium permanganate.

Before recommending this process the writers have investigated the influence of metals upon the accuracy of the assay and the influence of the variation of conditions. At the outset we found that the solution of the basic-oxalate in hydrochloric acid gave a very erratic end-point, and that the solution of basic-oxalate in sulphuric acid was too slow and gave a low result. We therefore used hydrochloric acid to dissolve the oxalate, neutralised with ammonia, and made quite acid with dilute sulphuric acid. This simple modification results in the titration being as direct and clear as an ordinary iron assay: one-half of one drop, or 0·05 c.c., showing the end-point.

We conduct an assay as follows:—

1. One grm. of the finely crushed pulp is attacked with 5 to 10 c.c. concentrated nitric acid, in a casserole covered with a watch-glass and evaporated to dryness on the hot plate. To the residue is added 5 c.c. nitric acid and the casserole replaced on the hot plate for a few minutes, breaking up the mass with a glass rod. We formerly filtered off the insoluble residue, but found it quite unnecessary. To the mass in the casserole 25 c.c. of water are added, and the contents emptied into a beaker and made up to about 100 c.c. with hot water.

2. To the bismuth solution 5 grms. of ammonium oxalate or oxalic acid are added and the whole boiled vigorously for five minutes. The beaker is removed from the hot plate, and in a minute or two the precipitate will completely settle. The supernatant is filtered off; with a rapid filter this is accomplished about as quickly as the liquid can be safely poured out of the beaker. The precipitate should be left as much as possible in the beaker.

3. The precipitate is boiled with two successive quantities (about 50 c.c.) of water; filtering through the same filter used in (2). These two boilings should entirely convert the bismuth oxalate into the basic salt when an ordinary ore of 10 per cent is being assayed. Should, however, the filtrate be acid to litmus-paper, boiling is continued until the final filtrate is neutral.

4. The basic oxalate on the filter is washed down to the point and 2 to 5 c.c. hydrochloric acid (1 acid to 1 water) are added, and the filtrate caught in the beaker containing

* A Paper read before the Royal Society of New South Wales, November 6, 1901.

the basic oxalate precipitate; the filter is washed with water twice. The basic oxalate is entirely dissolved by gentle warming. When entirely dissolved the solution is made up to 250 c.c. with hot water.

5. The hydrochloric acid in solution is neutralised with ammonia, about 2 c.c. should suffice. The resulting precipitate is re-dissolved by the addition of sulphuric acid (1 acid to 4 water) and an excess of a few c.c. added.

6. The solution is titrated at a temperature of from 70° C. to 100° C. The reaction between the oxalic acid and the permanganate is slow at first, but is instantaneous after a few drops have been added; the addition of permanganate is continued to a faint rose tint.

Precautions.—Insufficient nitric acid present in solution, before the addition of oxalate (operation 2), will result in the partial precipitation of the bismuth as basic nitrate and a low result will be obtained. Too much nitric acid must not be present; the deleterious effects of too much nitric acid can be obviated by adding a large amount of water. The bismuth oxalate must be entirely converted into basic oxalate or the results will be high. The normal oxalate is readily converted into the basic salt by repeated boilings with fresh quantities of water. When the filtrate is neutral to litmus-paper the reduction is complete. For the ordinary ore of 10 per cent two boilings generally suffice. The solution of the basic oxalate before titration must be perfect or the result will be low. Hydrochloric acid must not be used in dissolving up the ore. Nitric acid completely effects the solution of the bismuth in the ore.

Interferences.—Lead, iron, copper, zinc, arsenic, tellurium have no effect on the assay.

Some results obtained are as follows:—

Took Bismuth ore—			
1. Found 9·6% gravimetric, 9·58% volumetric.			
2. „ 4·49%	„ 4·49%	„	
3. „ 10·30%	„ 10·32%	„	
	Grms.		Grm.
4. 0·500 Bi containing	0·5 Pb	Found	0·5010 Bi
5. 0·500 „	„ 1·0 Pb	„	0·4968 „
6. 0·500 „	„ 1·0 Cu	„	0·4987 „
7. 0·500 „	„ 1·0 Zn	„	0·4994 „
8. 0·500 „	„ 0·7 Fe	„	0·5010 „
9. 0·500 „	„ 1·0 As_2O_3	„	0·4993 „
10. 0·500 „	„ 0·5 Sylvanite	„	0·5001 „

These results show that the method is quite accurate and suitable for all ordinary commercial work. Bismuth ores are, as a rule, quite free from large amounts of impurities, but the method is applicable to even very impure ores. By having an ample supply of hot water on hand the assay can be conducted very rapidly; half-an-hour will, as a rule, suffice to complete the assay; certainly no more than forty minutes will be required.—*Engineering and Mining Journal*, April 13, 1901.

ON THE RELATION OF THE VISCOSITY OF MIXTURES OF SOLUTIONS OF CERTAIN SALTS TO THEIR STATE OF IONISATION.*

By JAMES BARNES, B.A., Dalhousie College, Halifax, N.S.

The present paper is the result of a piece of work undertaken at the suggestion of Prof. MacGregor, for the summer vacation of 1899, the object being to find out in the case of mixtures of aqueous solutions of certain electrolytes with a common ion, whether or not it is possible, by the aid of the dissociation conception, to predict the viscosities of the mixtures, when sufficient data as to the viscosities and conductivities of the constituent solutions are available.

* *Transactions of the Nova Scotian Institute of Science*, x., p. 113.

The salts selected were sodium chloride, potassium chloride, barium chloride, sodium sulphate, potassium sulphate, and copper sulphate, the viscosities of simple solutions of these salts having been determined by Reyher (*Zeit. f. Phys. Chemie*, 1888, ii., 774), and Wagner (*Ibid.*, 1890, v., 31), and that of mixtures of them by Kanitz (*Ibid.*, 1897, xxii., 336), and extensive series of observations on the conductivity by Kohlrausch and by former students of Dalhousie College, being available. As will be seen below I found Kohlrausch's values of the conductivity sufficient for my purpose (Kohlrausch u. Holborn, "Leitvermögen der Elektrolyte," 1898, pp. 159, 160, tab. 2).

Professor MacGregor (*Trans. N. S. Inst. Sci.*, 1896-1897, ix., 219) has pointed out that, both on theoretical grounds and because of the way in which the ionisation coefficients and such physical properties as specific gravity, viscosity, &c., in general, vary with the concentration in simple solutions, it is to be expected that the value of any such property, for a simple solution which is so dilute that the dissociated and undissociated molecules may be regarded as without mutual action, will be expressed by the formula:—

$$P = P_w + k(1-a)n + l\,a\,n \quad . \quad . \quad . \quad . \quad (1)$$

where P is the numerical value of the property for the solution, P_w that of the same property for water under the same physical conditions, n the concentration expressed in gramme-equivalents per unit volume, a the ionisation coefficient of the electrolyte in the solution, and k and l constants, called ionisation constants.

He has further shown that the value of a property for a mixture of two electrolytes will be given in terms of the values of the ionisation constants as determined for the simple solutions, by the expression:—

$$P = P_w + \frac{1}{p}\left[\left(k_1(1-a_1)n_1 + l_1a_1n_1\right)\frac{v_1}{v_1+v_2} + \left(k_2(1-a_2)n_2 + l_2a_2n_2\right)\frac{v_2}{v_1+v_2}\right] \quad . \quad . \quad (2)$$

where the n's are the concentrations of the constituent solutions (the electrolytes being indicated by 1 and 2), the a's the ionisation coefficients of the respective electrolytes in the mixture, the v's the volumes of the constituent solutions, and p the ratio of the volume of the mixture to the sum of the volumes of the constituent solutions.

The application of the first expression to simple solutions is, as Prof. MacGregor has shown (*Trans. N. S. Inst. Sci.*, 1898-99, x., 61, footnote), of little theoretical interest; but that of the second to mixtures, because of its being based on the dissociation theory and involving no arbitrary constants, is of very considerable interest. It is the applicability of this expression (2) that the present paper is intended to test with mixtures of solutions of the above-mentioned salts.

The observations of Reyher, Wagner, and Kanitz were made on somewhat stronger solutions than those for which the above expressions might be expected to hold, but they were considered as probably sufficiently dilute to warrant the expectation of an approximate applicability of the expressions.

Simple Solutions.

For the determination of the ionisation constants in expressions (1) and (2), one must know the ionisation coefficients for the four solutions examined in the case of each salt by Reyher and Wagner. Unfortunately all the observations on the viscosity of these salts were made at 25° C., while all the available conductivity data, from which the ionisation coefficients are obtained, were at 18° C., and thus it was necessary either to reduce the viscosity values from 25° to 18° or the conductivity values from 18° to 25°. This latter reduction was carried out, as data for the former were not available. This involved

much work; because for the determination of the ionisation coefficients at 25°, it was necessary to obtain both the specific molecular conductivity at 25° and the specific molecular conductivity at infinite dilution for 25°.

Determination of the Specific Molecular Conductivity at Infinite Dilution for 25° C.

The value of the specific molecular conductivity at infinite dilution for 25° for each salt was obtained from Kohlrausch's value at 18° (Kohlrausch u. Holborn, *loc. cit.*, p. 200, tab. 8), by aid of Déguisne's data (*Ibid.*, p. 195, tab. 7). These data were employed in obtaining the specific molecular conductivity at 25° from the values at 18°, for the three weakest solutions given in Kohlrausch's and Déguisne's tables (*Ibid.*, pp. 159, 160, tab. 2); and the ratio—

$$\frac{\mu_{25} - \mu_{18}}{\mu_{18}}$$

was then determined, where μ_{25} and μ_{18} are the specific molecular conductivity at 25° and 18° respectively.

Table I. gives the values thus obtained. The concentrations are expressed in gramme-equivalents per litre, and the specific molecular conductivities in terms of this unit and of 10^{-4} times, Kohlrausch's new unit of conductivity (ohm^{-1} $cm.^{-1}$) (*Ibid.*, p. 1).

TABLE I.

Concentration.	Sp. mol. cond. at 18° C. (μ_{18}).	Sp. mol. cond. at 25° C. (μ_{25}).	$\frac{\mu_{25} - \mu_{18}}{\mu_{18}}$
	NaCl.		
0·0005	1085	1262	0·163
0·0002	1092	1270	0·163
0·0001	1097	1276	0·163
	KCl.		
0·0005	1283	1484	0·156
0·0002	1291	1494	0·157
0·0001	1295	1499	0·157
	$\frac{1}{2}BaCl_2$.		
0·0005	1183	1375	6·162
0·0002	1198	1394	0·163
0·0001	1205	1402	0·163
	$\frac{1}{2}K_2SO_4$.		
0·0005	1308	1516	0·159
0·0002	1327	1540	0·160
0·0001	1335	1549	0·160
	$\frac{1}{2}Na_2SO_4$.		
0·0005	1083	1266	0·169
0·0002	1096	1281	0·169
0·0001	1105	1292	0·169

As the ratio—

$$\frac{\mu_{25} - \mu_{18}}{\mu_{18}}$$

was thus found to be constant for the two most dilute solutions of every salt, and as these solutions are very dilute, this ratio may be assumed to approximately hold for infinite dilution. Observations on the conductivity of weaker solutions at different temperatures were not at hand; and the writer used the value of this ratio for the solution of concentration 0·0001 for the calculation of the specific molecular conductivity at infinite dilution for 25° C.

The following Table II. gives the values of the specific molecular conductivity at infinite dilution for 25° C. as thus obtained from the values at 18° C. In the case of copper sulphate this method could not be employed for want of data. A somewhat doubtful value obtained by Bredig was therefore used (*Zeit. f. Phys. Chem.*, 1894, xiii., 220). The conductivities are expressed as in Table I.

TABLE II.

Electrolyte.	Specific molecular conductivity at infinite dilution.	
	18° C.	25° C.
NaCl	1103	1283
KCl	1312	1519
$\frac{1}{2}BaCl_2$	1232	1433
$\frac{1}{2}K_2SO_4$	1350	1566
$\frac{1}{2}Na_2SO_4$	1141	1334
$\frac{1}{2}CuSO_4$	—	1423

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Extraordinary General Meeting, December 12th, 1901.

Prof. EMERSON REYNOLDS, Sc.D., F.R.S., President, in the Chair.

THE PRESIDENT stated that this meeting had been convened in accordance with a requisition signed by 128 Fellows of the Society, and that the following motions would be proposed:—

(1). On line 7 of By-law XI. (page 21, fifth line from top) to delete the words "the Council," and insert in their place the words "an Extraordinary General Meeting of the Society."

(2). To resolve that the Ordinary Meetings of the Society shall continue to take place, as heretofore, on Thursdays at 8 p.m.

Motion (1) was proposed by Mr. HEHNER, and seconded by Mr. PAKES.

Prof. FRANKLAND proposed, and Prof. RAMSAY seconded, the following amendment:—

"That this meeting desires to express its continued confidence in the Council as the Executive of the Society, as it has no reason to doubt that all the business of the Society, including the selection of days and hours of meeting, will be managed as heretofore in the interests of the Society as a whole and in accordance with the wishes of the majority of the Fellows."

After a discussion in which the following Fellows took part:—Dr. Divers, Mr. A. H. Allen, Mr. D. L. Howard, Professor Smithells, Dr. Lewkowitsch, Professor Dewar, Professor Warington, and Mr. Phipson Beale, K.C., the PRESIDENT asked for a show of hands, and declared the amendment carried. A poll having been demanded, the PRESIDENT announced that it would be taken by means of cards having "For" and "Against" printed on them, with a space for the signature of the Fellow voting, one of these words to be deleted. Mr. Heycock, Dr. Hewitt, Dr. Shields, and Dr. Travers, having been appointed scrutators,

The PRESIDENT announced that the votes were—For the amendment, 128; against, 120.

The PRESIDENT then declared the following carried:—

"That this meeting desires to express its continued confidence in the Council as the Executive of the Society, as it has no reason to doubt that all the business of the Society, including the selection of days and hours of meeting, will be managed as heretofore in the interests of the Society as a whole, and in accordance with the wishes of the majority of the Fellows."

Ordinary Meeting, December 19th, 1901.

Prof. J. EMERSON REYNOLDS, F.R.S., President, in the Chair.

Messrs. Kiddell, Larter, Harding, and H. C. T. Gardner were formally admitted Fellows of the Society.

The SECRETARY read the following Address which was presented on November 24th to Professor Berthelot in the name of the Chemical Society by the President, who was accompanied by Dr. Gladstone and Professor Ramsay, as the representatives of the Society :—

To M. MARCELLIN BERTHELOT, *Senator, late Minister for Foreign Affairs, Member of the Institute.*

SIR,—On behalf of the President, Officers, Council, and Fellows of the Chemical Society, we beg to offer you, the Senior Foreign Member of our Society, our heartiest congratulations on the occasion of the Fiftieth Anniversary of your first scientific publication.

The Chemical Society has recently received the first volumes of your monumental work, yet to be completed, including the great number of papers which you have contributed, during fifty years of scientific activity, to the Chemistry of the hydrocarbons, and their methods of formation.

There is, however, scarcely any department of Chemical Science in which you have not worked as a pioneer with distinguished results, whilst to you is largely due the foundation of a new branch of the Science, namely, Thermo-Chemistry.

Alike in the history of our Science, and in its applications to Agriculture and Industry, you have made brilliant contributions.

We trust that for many years you may possess the health and strength to continue those investigations which have made your name famous throughout the Scientific world.

Signed, on behalf of the Chemical Society,
J. EMERSON REYNOLDS, President.
WILLIAM A. TILDEN, Treasurer.
WYNDHAM R. DUNSTAN, ALEXANDER SCOTT, } Secretaries.
RAPHAEL MELDOLA, Foreign Secretary.

November 22nd, 1901.

Day and Hour of Meeting.

The PRESIDENT announced that at the Meeting of Council held this afternoon, the following resolution was unanimously adopted :—

"That having regard to the discussion which took place at the Extraordinary General Meeting on December 12th, the Council resolves to make the experiment of holding the Ordinary Meetings, as far as possible, alternately at 5.30 p.m. on Wednesdays, and at 8 p.m. on Thursdays, from January, 1902, until the end of the present session in June, instead of holding meetings on Wednesdays at 5.30 p.m. only during the period from January to March, 1902, as stated in the resolution of July 4th, 1901, which is hereby rescinded,"

And that a Committee had been appointed, consisting of the President, the Treasurer, the Senior Secretary, Dr. Armstrong, Professor Dobbie, and Dr. Forster, to arrange the exact dates on which future meetings during the session should be held.

The next Ordinary Meeting of the Society will be held on Thursday, January 16th, at 8 p.m.

It was also announced that Sir Henry Roscoe had presented a plaster cast of the bronze portrait on Bunsen's tomb in Heidelberg, and that a photograph of a bas-relief of Professor Julius Thomsen had been presented to the Society by Mr. Harald Faber.

Certificates were read for the first time in favour of Messrs. Evelyn Andros de la Rue, 52, Cadogan Square, S.W.; Keith Benham Benham, Deans Hall, Stafford; William Dennis, Ocean Road Pharmacy, South Shields; Paul Haas, 11, Westbourne Park Road, W.; Eugene Edwin Hennesey, Bigods, Dunmow, Essex; William Holdsworth Hurtley, St. Bartholomew's Hospital, E.C.; David Smith Jardin, Rathgar House, Rathgar; Harry Lucas, 1, St. Agnes' Place, Kennington Park, S.E.; John Ross MacKenzie, 31, Bailey Street, Ton-Pentre, Glam.; William Maitland, 236, Brookhill, Sheffield; Francis Martin, 64, Samuel Street, Woolwich; Francis Hylton Molesworth, Turramurra, Sydney, N.S.W.; Alfred Holley Mundey, 17, St. Margaret's Road, Plumstead; Robert Eley Blake Smith, 93, Upper Richmond Road, Putney, S.W.; William Southworth, County Council Farm, Hutton, Preston, Lancs.

Of the following papers, those marked * were read:—

*171. "*Corydaline.* Part VII. *The Constitution of Corydaline.*" By J. J. DOBBIE, D.Sc., M.A., and A. LAUDER, B.Sc.

The decomposition products of corydaline have been studied by the authors in greater detail with the following results :—The acid, $C_6H_4N(CO_2H)_3$, obtained by oxidising corydaline (*Trans.*, 1897, lxxi., 657), is, by long-continued heating with potassium permanganate in alkaline solution, converted into 2 : 3 : 4 : 6-pyridinetetracarboxylic acid. The acid, $C_6H_4N(CO_2H)_3$, must therefore either be 2—CH_3 3 : 4 : 6-pyridinetricarboxylic acid or 2—CH_3 4 : 5 : 6-pyridinetricarboxylic acid. From what follows, the former formula is probably the correct one.

Corydilic acid, $C_{12}H_6N(OCH_3)_2(CO_2H)_3$ (*Trans.*, 1897, lxxi., 657), is soluble in hydrochloric acid, and under certain conditions forms an unstable hydrochloride. When boiled with potassium permanganate, it is split up into a mixture of meta-hemipinic acid and the acid $C_6H_4N(CO_2H)_3$.

When corydic acid (*Trans.*, 1897, lxxi., 657), $C_{14}H_9N(OCH_3)_2(CO_2H)_2$, is oxidised with permanganate at the ordinary temperature, it yields a yellow, dibasic acid, $C_{12}H_9N(OCH_3)_2(CO_2H)_2$, which contains the two methoxyl groups of corydic acid.

Mild oxidising agents remove four atoms of hydrogen from corydaline and convert it into dehydrocorydaline, an intensely yellow coloured base (*Trans.*, 1897, lxxi., 657). Berberine is also a yellow coloured base, but readily takes up four atoms of hydrogen, forming colourless tetahydroberberine. Corydaline therefore corresponds to tetrahydroberberine and dehydrocorydaline to berberine.

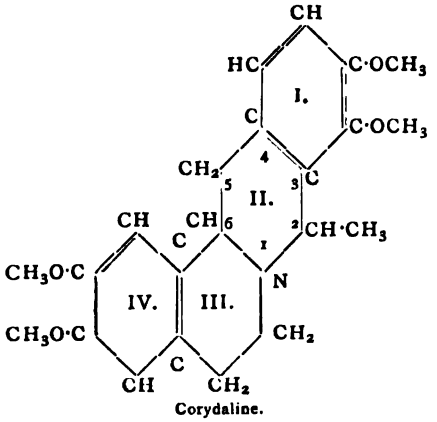
Corydaline (colourless).	Tetrahydroberberine (colourless).
$C_{22}H_{27}NO_4$.	$C_{20}H_{21}NO_4$.
Dehydrocorydaline (yellow).	Berberine (yellow).
$C_{22}H_{23}NO_4$.	$C_{20}H_{17}NO_4$.

The four oxygen atoms of corydaline are all present in methoxyl groups (*Trans.*, 1892, lxi., 605). When oxidised with potassium permanganate at 100°, corydaline yields a mixture of hemipinic and metahemipinic acids, together with corydaldine, $C_{11}H_{13}NO_3$, a neutral substance of which the yield is larger when the oxidation takes place at the ordinary temperature. This substance has been shown to be an *iso*quinoline derivative.

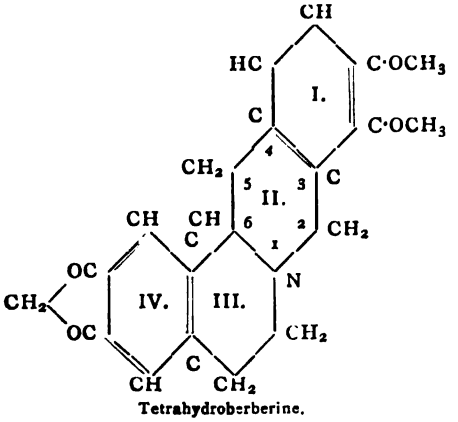
When oxidised with dilute nitric acid, corydaline, $C_{22}H_{27}NO_4$, is first converted into dehydrocorydaline, $C_{22}H_{23}NO_4$; further oxidation splits off one of the benzene rings, and yields corydic acid, a yellow, crystalline substance (*Trans.*, 1897, lxxi., 657). Corydic acid, in turn, when oxidised at 100° with permanganate, yields meta-hemipinic acid, the acid $C_6H_4N(CO_2H)_3$, and the highly insoluble, colourless corydilic acid, $C_{12}H_6N(OCH_3)_2(CO_2H)_3$, which, on further oxidation with permanganate, is entirely resolved into meta-hemipinic acid and the acid $C_6H_4N(CO_2H)_3$.

As the acid $C_6H_4N(CO_2H)_3$ contains six atoms of carbon in addition to the carbon atoms of the carboxyl groups, it cannot be derived from the *iso*quinoline nucleus which has no methyl group attached to it, but must represent a fourth ring, and the nitrogen atom must, as in the case of berberine, be common to two rings.

There are thus four rings in corydaline. The following formula satisfactorily explains the formation of all the derivatives of corydaline which have been examined :—

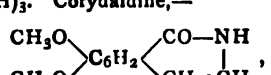


Corydaline.



Tetrahydroberberine.

By oxidation, rings I. and IV. would yield hemipinic and meta-hemipinic acids respectively; ring II., the acid $C_6H_4N(CO_2H)_3$. Corydaldine,—

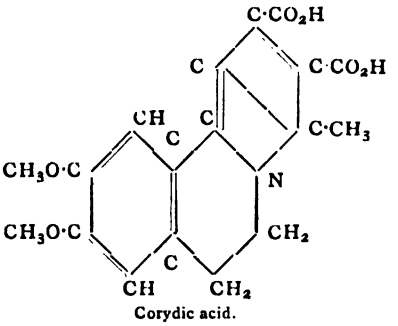


contains rings III. and IV., and would result from the oxidation of corydaline, just as ω-amidoethylpiperonylcarboxylic anhydride results from the oxidation of berberine. Corydic acid would be formed by the destruction of ring I., and corydilic acid from corydic acid by the oxidation of ring III. The formulæ of the last-mentioned acids are therefore as given in next column.

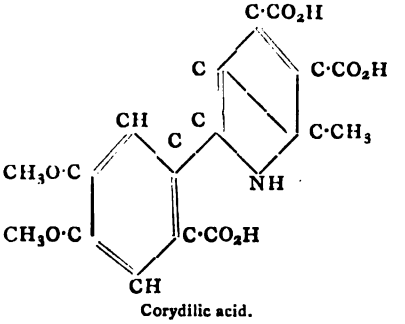
A comparison between the formulæ of corydaline and tetrahydroberberine explains the resemblance between the two substances and their oxidation products. By oxidation, ring I., both in berberine and corydaline, yields hemipinic acid; ring IV. in berberine yields hydrastic acid, corresponding to meta-hemipinic acid from corydaline. ω-Amidoethelypiperonylcarboxylic anhydride, corresponding to corydaline, is derived from rings III. and IV., and berberonic acid, $C_5H_2N(CO_2H)_3$ [$(CO_2H)_3$=3 : 4 : 6] corresponding to the acid $C_6H_4N(CO_2H)_3$, from ring II. in corydaline.

Berberonic acid is derived from ring II., and not from ring III., which would yield carbocinchomeronic acid. This point is not dealt with by Perkin, but incidentally affords confirmation of the correctness of his formula.

The ease with which corydaline is oxidised to dehydrocorydaline is explained in the same way as in the case of the oxidation of tetrahydroberberine to berberine, the hydrogen atoms attached to the carbon atoms 2, 5, and 6 of ring II. being removed on oxidation, and a link



Corydic acid.



Corydilic acid.

established between the atoms 2 and 5, and a second link between 5 and 6.

The above formula for corydaline, which accounts for its decomposition products and explains the analogies with berberine, involves the adoption of the constitution 2—CH_3 : 3 : 4 : 6 for the acid $C_6H_4N(CO_2H)_3$; the alternative formula would leave the resemblance to berberine unexplained.

*172. "*The Relation of Corydaline to Berberine; the Oxidation of Berberine with Nitric Acid.*" By J. J. Dobbie, D.Sc., M.A., and A. Lauder, B.Sc.

In the previous abstract it is shown that the constitution of corydaline can be expressed by a formula similar to one of the formulæ proposed by Perkin for berberine.

Assuming the correctness of the formula for corydaline, the absence of compounds corresponding to berberal and berberilic acid (Perkin, *Trans.*, 1890, lvii., 992) from amongst the decomposition products of corydaline, is explained, since all these substances contain an atom of oxygen united to the carbon atom 2 in ring II. (see previous abstract), where owing to the presence of the methyl group it is impossible to introduce an oxygen atom into corydaline derivatives. If the view of the relation of the two substances expressed in the formulæ is correct, the first of Perkin's alternative formulæ (Perkin, *Trans.*, 1890, lvii., 992) must be adopted. In his second formula, a double bond is shown between the carbon atoms 2 and 5 of ring II. No double bond in this position is possible in dehydrocorydaline on account of the presence of the methyl group, and the analogy between the two substances breaks down if we suppose the double bond in ring II. to occupy different positions. Whilst the absence of certain decomposition products is thus satisfactorily accounted for, the formation of corydic acid from corydaline suggests the possible formation of a similar acid from berberine. By oxidising berberine with dilute nitric acid in exactly the same way as was followed in the preparation of corydic acid (*Trans.*, 1897, lxxi., 657), an acid, $C_{16}H_{11}NO_6$ (m.p. 285°), was obtained closely resembling corydic acid in properties and obviously bearing to berberine the same relation as corydic acid to corydaline, This acid is very difficultly soluble in hot water; it dissolves in sodium

hydroxide, forming a blood-red solution. It is dibasic, almost all its salts are soluble, a notable exception being the acid silver salt. It contains no methoxyl groups, hence (as in the case of corydaline) it is formed by the destruction of ring I. By oxidation with potassium permanganate, it yields berberonic acid, $C_5H_2N(CO_2H)_3$ [$(CO_2H)_3 = 3:4:6$], and ω-amidoethylpiperonylcarboxylic anhydride. The name *berberidic* is proposed for this new acid.

*173. "*The Magnetic Rotation of some Polyhydric Alcohols, Hexoses, and Disaccharoses.*" By W. H. PERKIN, Sen., Ph.D., F.R.S.

The object of this investigation was to see if any clue could be obtained from the magnetic rotation of the sugars as to the cause of their bi- or multi-rotation. Some of the polyhydric alcohols were also examined in order that data might be obtained from which to calculate the probable values for the various sugars.

From the rotations obtained for glucose and for fructose, it was found that the suggestion that bi-rotation was due to hydration was untenable. The magnetic rotations are too low for substances possessing the aldehydic and ketonic constitutions usually assigned to these sugars, but they correspond with substances containing an oxygen atom linked as in ethylene oxide or the lactones, all of which give low numbers. This agrees with the suggestion of Lowry (*Trans.*, 1899, lxxv., 215), that when in solution and having undergone the largest amount of change, glucose exists chiefly in an isomeric form for which he gave two formulæ, of which the more probable one, judging from the magnetic rotation, may be represented thus:—

$$CH_2OH\cdot CH\cdot OH\cdot CH\cdot CH\cdot OH\cdot CH\cdot OH\cdot CHOH$$
$$\text{|}\!\!-\!\!-\!\!-\!\!-\!\!-\!\!-\!\!-\!\!-\!\!-\!\!-\!\!-\!\!-\!\!-\text{|}$$
$$O$$

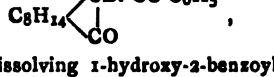
This is the formula originally proposed by Tollens for dry glucose (*Ber.*, 1883, xvi., 92). When slightly modified it can also be used to represent fructose in an isomeric condition, equally consistent with the magnetic rotation of this substance in solution.

Saccharose may be regarded as built up from glucose and fructose, both being in their isomeric forms, with elimination of water, its constitution being that proposed by E. Fischer (*Ber.*, 1893, xxvi., 2405), which is a slight modification of that suggested by Tollens (*Ber.*, 1883, xvi., 923).

Maltose and lactose, which possess both bi-rotation and reducing power, appear to have analogous structures, the formula proposed by Fischer for lactose (*loc. cit.*) being practically applicable to both, but their constitution is different from that of saccharose. Hence maltose and lactose are formed from a molecule of glucose or of galactose in the isomeric condition, *plus* a molecule of glucose in the ordinary or aldehydic form, *minus* water, the aldehydic part undergoing isomeric change to a greater or less extent when these disaccharoses are dissolved in water; in this way the possession of bi-rotation by these substances may be explained.

*174. "*Stereoisomeric Halogen Derivatives of α-Benzoylcamphor.*" By M. O. FORSTER and Miss F. M. G. MICKLETHWAIT.

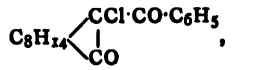
α'-Benzoyl-α-bromocamphor—



prepared by dissolving 1-hydroxy-2-benzoylcamphene in glacial acetic acid containing sodium acetate (1½ mols.) and adding a solution of bromine (1 mol.) in acetic acid, crystallises from light petroleum in large transparent, six-sided prisms, and melts at 114°; dissolved in benzene it has $[\alpha]_D = -10{\cdot}0°$ and in chloroform $[\alpha]_D = +10{\cdot}3°$.

α-Benzoyl-α'-bromocamphor is formed in preponderating amount when potassium hypobromite acts on hydroxybenzoylcamphene dissolved in potash; it crystallises from hot alcohol in transparent, rectangular plates melting at 214°, and has $[\alpha]_D = -53{\cdot}2°$ in benzene and $[\alpha]_D = -19{\cdot}3°$ in chloroform. Hydrogen bromide converts the low melting isomeride into the less readily fusible modification.

α'-Benzoyl-α-chlorocamphor—

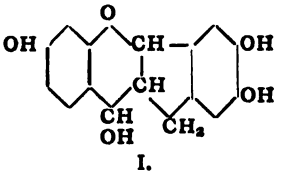


obtained by the action of sodium hypochlorite on hydroxybenzoylcamphene, melts at 88°, and has $[\alpha]_D = -27{\cdot}9°$ in chloroform.

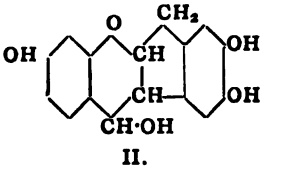
α-Benzoyl-α'-chlorocamphor is produced in the same way and separated by means of its sparing solubility in alcohol; it melts at 219° and has $[\alpha]_D = +26{\cdot}2°$ in chloroform.

*175. "*Brasilin and Hæmatoxylin.* Part VI. *The Constitution of Brasilic Acid of Brasilin and of Hæmatoxylin.*" By W. H. PERKIN, Jun.

In Part I. of this research (A. W. Gilbody, W. H. Perkin, jun., and J. Yates (*Trans.*, 1901, lxxix., 1401), it was argued that since trimethylbrasilin on oxidation with permanganate yields 2-carboxy-5-methoxyphenoxyacetic acid and meta-hemipinic acid, the constitution of brasilin must be represented by one of the following formulæ:—



I.



II.

In order to decide between these two formulæ, the author has submitted brasilic acid to a detailed examination and has obtained results which show clearly that formula I. must be accepted as representing the constitution of brasilin.

Brasilic Acid, $C_{12}H_{12}O_6$, is produced by the oxidation of trimethylbrasilin with permanganate, but the yield is only 0·7 per cent; it is a monobasic acid, the *silver* salt having the formula $C_{12}H_{11}AgO_6$. The sodium salt $C_{12}H_{11}NaO_6$, is comparatively sparingly soluble and crystallises in glistening plates.

Since brasilic acid contains one methoxyl group, its formula may be written $MeO\cdot C_{10}H_8O_3\cdot CO_2H$, and as on fusion with potash it yields a substance which with ferric chloride gives a violet colouration, it is evidently derived from the resorcyl nucleus of brasilin. Furthermore, it yields an oily oxime, $MeO\cdot C_9H_8O_2(C{:}NOH)CO_2H$, and when reduced with sodium amalgam it is converted into the *lactone* of *dihydrobrasilic acid*, $C_{12}H_{12}O_5$, which melts at 144° and is very sparingly soluble in ether.

From this behaviour, it follows that brasilic acid contains a carbonyl group, and that this is probably in the γ-position in relation to the carboxyl group.

When warmed with sulphuric acid, brasilic acid loses one molecule of water and is converted into *dehydrobrasilic acid*, $C_{12}H_{10}O_5$, a monobasic acid, which is very sparingly soluble in water, melts at 197°, and when treated with permanganate in the cold is oxidised with formation of *p*-methoxysalicylic acid, $MeO\cdot C_6H_3(OH)\cdot CO_2H$.

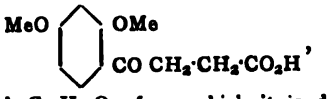
Hydroxylamine converts dehydrobrasilic acid into an oxime, $C_{12}H_{11}NO_5$, which melts at 172°. The acid, therefore, still contains a carbonyl group. When digested

with baryta water, it is readily decomposed with formation of formic acid and a new acid, $C_{11}H_{12}O_5$.

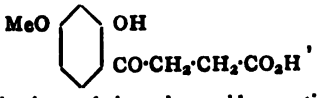
$$C_{12}H_{10}O_5 + 2H_2O = H{\cdot}CO_2H + C_{11}H_{12}O_5.$$

This new acid crystallises from water in colourless needles, melts at 155°, and gives, in aqueous solution, an intense violet colouration with ferric chloride; when heated with sodium methoxide and methyl iodide it yields a methyl derivative, $C_{12}H_{14}O_5$, which melts at 147°.

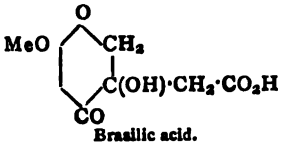
In conjunction with Mr. E. Ormerod, the author has succeeded in synthesising this latter by treating dimethylresorcinol in the presence of aluminium chloride with the ester of the half-chloride of succinic acid, $COCl{\cdot}CH_2{\cdot}CH_2{\cdot}CO_2Et$; the metbyl derivative, $C_{12}H_{14}O_5$, must therefore be 2 : 4-dimethoxybenzoylbutyric acid—



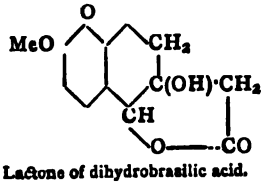
and the acid, $C_{11}H_{12}O_5$, from which it is derived by methylation, is 2-hydroxy-4-methoxybenzoylbutyric acid—



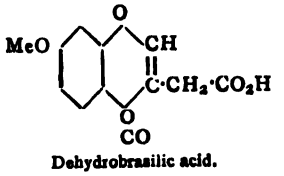
The constitutions of the other acids mentioned above are now established, and are as follows :—



Brasilic acid.



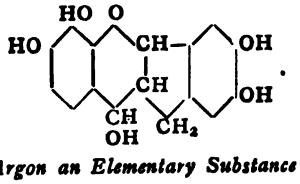
Lactone of dihydrobrasilic acid.



Dehydrobrasilic acid.

Since of the two formulæ for brasilin given above, formula I. alone accounts, in a simple manner, for the formation of brasilic acid, it appears to the author that this must be accepted as representing the constitution of brasilin.

It has already been shown (*Proc.*, 1900, xvi., 107) that tetramethylhæmatoxylin, on oxidation with permanganate, gives products which are exactly similar to those obtained from trimethylbrasilin, and there can, therefore, scarcely be a doubt that the constitutional formula of hæmatoxylin is—



176. "*Is Argon an Elementary Substance?*" By G. Martin.

Some eighty distinct elementary substances are now recognised. These are assumed to be composed of different kinds of matter because each one has *chemical properties* peculiar to itself alone. Physical properties are of little value in deciding whether different substances are merely allotropic modifications of one kind of elementary matter, or whether they are distinct elements. The only certain test lies in the *chemical nature* of the compounds produced when the elements unite with other elements.

Elements often resemble each other so strongly in their physical nature that it is almost impossible to distinguish between them. For example, the new radio-active elements are so similar to bismuth, barium, and titanium, that many chemists still hesitate to believe them elementary. The rare earths are composed of elements so much alike that it is with the utmost difficulty they can be distinguished from one another.

But all these substances have one characteristic which stamps them as elements—*each produces its own peculiar set of compounds*. This characteristic is absent from argon and its companions; therefore, in order to demonstrate their elementary nature, Ramsay had to fall back on their physical properties—the most untrustworthy of all methods.

These properties stamp the new gases as a distinct class of elements; but they do not, and cannot, prove that each "element" is a single substance and not a *group* of closely related elements. For instance, were *argon* a *mixture* of 3 monatomic gases of like nature, and of which the atomic weights differed from each other by a fraction of a unit—as do those of nickel and cobalt—it would be impossible to distinguish the mixture from an element. It would answer to exactly the same physical tests, and could not be resolved into its compounds by any *physical methods* except with the utmost difficulty. It takes many thousands of fractionations to distinguish between two rare earths, which not only give different chemical compounds, but are far more unlike chemically than would be 3 *monatomic inert gases*.

We are peculiarly liable in the eighth group to meet with neighbouring elements of almost identical atomic weight and properties. For instance, in series 4 of the Periodic Table, as we advance with increasing atomic weight from potassium to nickel, the resemblance between successive elements steadily increases; potassium and calcium, for example, are quite unlike each other, but vanadium, chromium, manganese, and iron are all strikingly analogous, while the two last elements of the series, cobalt and nickel, have almost identical atomic weights, and resemble each other in a most remarkable degree.

In many of the other series, also, we end up in the eighth group with elements so very similar that they are classed as "triads"; for example, ruthenium, rhodium, and palladium; osmium, iridium, and platinum; the atomic weights of these triads are in every case very close together.

If each of the "elements" of Ramsay really consists of three allied substances, the eighth group of the periodic table would lose altogether its peculiar "triad" character, and split up into three distinct groups—a possibility which of late years has been running through many minds.

177. "*The Action of Phosphorus Trithiocyanate on Alcohol.*" By Augustus Edward Dixon, M.D.

Lössner states in a brief preliminary note (*Journ. Prakt. Chem.*, 1873, [2], vii., 474) that phosphorus trichloride, acting upon an alcoholic solution of potassium thiocyanate, produces a substance crystallising in fine needles, and having the formula, $C_8H_{18}N_4S_4O$.

From results obtained by the author in his study of phosphorous and phosphoryl 'thiocyanates,' recently commenced (*Trans.*, 1901, lxxix., 541), it seemed probable that Lössner's compound, if really formed, must have originated through some decomposition of the phosphorus trithiotriurethane, $P(NH{\cdot}CS{\cdot}OEt)_3$, which might be expected to result from the union of phosphorus trithiocyanate, $P(SCN)_3$, or $P(NCS)_3$, with the alcohol used as

solvent; to ascertain if this was the case, the two latter substances were caused to interact directly, in presence of dry benzene.

Phosphorus trithiotriurethane could not be identified; neither could Lossner's compound; thiocyanic acid was expelled, and the residual liquid was eventually resolved into (1) an acid oil, containing phosphorus, and (2) *iso*persulphocyanic acid.

Phosphorus trichloride interacted violently with alcoholic potassium thiocyanate: potassium chloride was precipitated, thiocyanic acid is given off, and from the residual oily liquid, after concentration, *iso*persulphocyanic acid was deposited: no sign could be got of the presence of the compound $C_8H_{18}N_4S_4O$.

The author supposes the *iso*persulphocyanic acid, which is formed in small relative amount in either process, to be produced through the interaction of the mineral acid with the thiocyanic acid simultaneously liberated.

Benzyl alcohol interacts spontaneously with phosphorus trithiocyanate, but, as in the case of ethyl alcohol, no evidence could be obtained of the existence of a salt of phosphorus trithiotricarbamic acid, $(P(NH\cdot CS\cdot OH)_3$ if such compounds are formed at all in these interactions, it is probable that, like their congeners of the thiocarbamidic class, they very readily undergo hydrolysis.

(To be continued).

NOTICES OF BOOKS.

Jahrbuch der Elektrochemie. VII. Jahrgang. By Dr. W. NERNST and Dr. W. BORCHERS. Halle a. S.: Wilhelm Knapp. 1901.

THE editors of this annual publication have to record many notable advances both in the applied and pure branches of electrochemistry. The task of summarising the progress of the science has been rendered easier owing to the many international congresses held in connection with the Paris Exhibition, where for the first time electrochemistry was allotted a place of its own among other scientific industries; these unusually favourable circumstances have induced Dr. Borchers, who is responsible for the greater part of the section of the book devoted to applied electrochemistry, occasionally to recapitulate many already known facts, so that we have in the case of some of the elements a short review of the present state of our knowledge of the various methods, both new and old, of preparing them. Wherever possible, detailed illustrated accounts of innovations in commercial processes are given for the more important elements and compounds, together with lists of German, English, and American patents, which should render the annual most useful to the inventor and the manufacturing chemist.

The year 1900 saw great progress in the application of electrochemical methods to the preparation of organic bodies, by which means many already known substances are prepared more easily than by purely chemical methods, and also various new substances have been obtained. More important, perhaps, is the extension of electrical methods of reduction and oxidation which lead to most interesting results. The section of the book devoted to organic chemistry contains accurate and full accounts of all that has been done in this respect, especially with regard to the reduction of aromatic nitro-bodies, on which Haber and Sonneborn, amongst others, have done much research.

Two indexes are given, besides lists of publications of the year 1900 in chemistry and allied subjects, and the book is well illustrated. In view of the increasing importance of electrochemical methods of preparation, such an annual as this should prove a valuable book of reference for the physical chemist and the advanced technical student.

Annuaire Pour l'an 1902. Publié par le Bureau des Longitudes. Paris: Gauthier-Villars.

WE have received this annual for 1902. It is a small compact volume containing a large amount of matter which will be very useful both to the engineer and the student of science. Among the notices included in the volume we may especially direct attention to that of M. Cornu on "Polyphase Currents," that of M. Poincaré on "Telegraphing without Wires," and that of M. Guyou on "The Decimal Division of the Circumference." The volume is in 16mo, and contains about 850 pages. It is furnished with a good index, as well as a complete table of contents. The price is 1fr. 50c.

OBITUARY.

SIR HENRY GILBERT.

IT is with great regret that we record the death of Sir Henry Gilbert, the well-known agricultural chemist, which occurred on December 23rd, 1901, at his residence at Harpenden, St. Albans.

Sir Henry was born at Hull in 1817, and was the son of the Rev. Joseph Gilbert. Shortly after leaving school he was the victim of an accident from a shot-gun, which destroyed the sight of one eye, besides impairing his health for some time. Later on, he went to Glasgow University, where he made chemistry his principal study; thence he proceeded to University College, London, under Professor Graham. He also worked in the laboratory of Dr. A. T. Thomson, where he met his future collaborator, John Bennet Lawes. He next went to Germany to the University of Giessen, under Liebig, and there he took the degree of Ph.D. On returning to London in 1840 Dr. Gilbert went as assistant to his old teacher, Professor A. T. Thomson, at University College.

In the year 1843 he became associated with John Lawes, and assumed control of the chemical laboratory at the Rothamsted Experimental Station, Harpenden, where he spent practically the whole of the remainder of his life in work that has turned out to be of such importance to agricultural chemistry, most of which was given to the world in papers under the joint authorship of Sir John Lawes and himself.

In the year 1841 Sir Henry (then Dr.) Gilbert was elected a Fellow of the Chemical Society, within three months of its foundation. In 1882 and 1883 he held the office of President of the Society. In 1860 he was elected a Fellow of the Royal Society, and in 1867 he was awarded one of the Royal Medals.

Sir Henry Gilbert held several Professorships, and was a member of many agricultural and other learned societies, besides holding many honorary degrees.

MR. HENRY GEORGE MADAN.

WE have also to record with much regret the death of Mr. Henry George Madan, the Senior Fellow of Queen's College, Oxford, which occurred at Bearland House, Gloucester, on Sunday evening, the 22nd of December, 1901.

Mr. Madan was born on September 6th, 1838, and was therefore sixty-three years of age. He was educated at Marlborough College, whence he obtained an open exhibition at Corpus Christi College, Oxford, in 1857. He obtained a second-class at Classical Moderations in 1858, and a first-class in Natural Science in 1861.

In the year 1862 Mr. Madan was elected Fellow of Queen's College, and for some time acted as assistant to Sir Benjamin Brodie, the then Professor of Chemistry.

He was for twenty years the Head of the Science Department at Eton. Mr. Madan was a Fellow of the Chemical Society, and was the joint author with Mr. A. G. V. Harcourt, of Christchurch, of "Exercises in Practical Chemistry," and of several other smaller works on chemistry and physics. Some months ago Mr. Madan was knocked down by a railway truck in the docks at Gloucester, and suffered injuries which necessitated the amputation of his right arm, and affected his health in a manner which he never got over. His death will be regretted by a large circle of sympathetic friends.

CORRESPONDENCE.

NIOBIUM IN TUNGSTEN MINERALS AND METALS.

To the Editor of the Chemical News.

SIR,—Of the raw materials of steel-making tungsten compounds only are definitely known to sometimes contain niobium. An analysis by Bodenbender showed the presence of 1·22 per cent niobic oxide in wolframite, and the element is said to find its way into metallic tungsten powders.

Many who have to make analyses of these materials would be pleased to learn whether any reliable means of detecting and estimating small amounts of niobium are known. One's quest in this direction is somewhat complicated by the element being sometimes called niobium and sometimes columbium.

Osborne's paper "On the Quantitative Estimation of Niobium (CHEMICAL NEWS, liii., 43) calls attention to the presence of small amounts of fluorides entirely preventing the hydrogen peroxide colour with titanium solutions, which is recalled by Hillebrand's warning against fluoriferous hydrogen peroxide (CHEMICAL NEWS, lxxxiv., 311; and lxxii., 158).—I am, &c.,

HARRY BREARLEY.

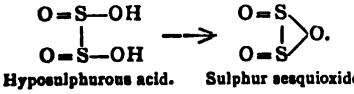
Bower Road, Sheffield.

THE CONSTITUTION OF SULPHUR SESQUIOXIDE.

To the Editor of the Chemical News.

SIR,—Sulphur sesquioxide is generally represented by the formula S_2O_3; I think the facts show that it should be formulated S_4O_6.

Sulphur sesquioxide is by some looked upon as the anhydride of hyposulphurous acid,—



Hyposulphurous acid. Sulphur sesquioxide.

But such a view must be considered untenable, for, by hydrolysis, it neither yields hyposulphurous acid nor its products of decomposition.

It is also impossible to devise any satisfactory structural formula for S_2O_3 which will represent its formation from sulphur trioxide and sulphur, and its (easy) decomposition into sulphur dioxide and sulphur.

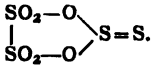
For the formula S_4O_6, I put forward the following facts:—

SO_2 is a gas,
SO_3 is practically a liquid,
S_2O_7 is hardly a solid,
and S_2O_3 is a crystalline solid.

The increase in melting-point cannot wholly depend upon the degree of oxidation, for then sulphur sesquioxide would have the lowest melting-point. Neither can it wholly depend upon the degree of sulphonation, for then SO_2, for instance, would have a higher melting-point than SO_3. It might be urged that sulphur sesquioxide had a h gh melting-point because it had two atoms of sulphur; but, now, SO_3 has a higher melting-point than SO_2, consequently the oxygen does to some extent increase the melting-point; and, upon the same reasoning, if sulphur sesquioxide were S_2O_3, it should have a lower melting-point than S_2O_7. In fact, the only way we can account for the higher melting-point of sulphur sesquioxide is by assigning to it the formula S_4O_6; it will then fall in its right position:—

SO_2	=	3 atoms.
SO_3	=	4 ,,
S_2O_7	=	9 ,,
S_4O_6	=	10 ,,

As to its structural formula, the following may be put forward:—



This well represents its formation from sulphur trioxide and sulphur, and also the ease with which it decomposes into sulphur dioxide and sulphur. A little consideration will also show that it represents the products formed by the hydrolysis of sulphur sesquioxide (sulphuric, sulphurous, thiosulphuric, pentathionic acids, and sulphur), but space forbids the graphical representation which is necessary.—I am, &c.,

A. LIONEL LANDAU.

20, Highbury New Park, N.,
December 23, 1901.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxiii., No. 24, December 9, 1901.

Researches on Radium.—H. Berthelot.—The author obtained a specimen of radium from M. Curie. He especially examined two interesting points; the part played by phosphorescence, and the order of greatness of the energies brought into action by the intervention of this new element. He operated at temperatures of 10° and 100°, always under atmospheric pressure, and varying the time of the action of radium. In each case three experiments were performed with iodic acid, one a blank, one in presence of a tube containing radium, and susceptible to the action of phosphorescence, and, finally, an experiment in presence of the same tube surrounded by black paper to intercept all influence which might be due to phosphoresence. An examination of the action produced was made at the end of definite periods of time.

Radio-activity of Uranium.—Henri Becquerel.—The radio-activity of uranium has been proved not to be constant. M. Giesel in particular showing that after certain treatments preparations of uranium became less active, and Sir W. Crookes, by fractional crystallisations, obtained inactive uranium nitrate. Reviewing these and various other researches on this subject, the author puts forward an hypothesis which apparently agrees with observed facts. If the emission of deviable rays identical with kathode rays were the cause of the emission of non-deviable radiation, which is so much like X rays, this spontaneous emission could be compared to the evaporation of an odorous body, the radio activity resembling a known phenomenon. The dissipated energy would be

given out from the active body itself, but the corresponding loss of weight would be too feeble to be observed.

Alloys of Aluminium and Magnesium.—O. Boudouard.—A previous research on the fusibility of alloys of aluminium and magnesium enabled the existence of two definite compounds, $AlMg_2$ and AlMg, to be predicted. By utilising the known constants, by a microscopic examination, and also by chemical methods, the author was able to isolate, in addition to the others, the compound Al_4Mg. M. Le Chatelier's method was used, but the results were not very satisfactory in general. However, crystals of Al_4Mg were separated by attacking the metallic mixture with a 10 per cent solution of hydrochloric acid. By varying the proportions of aluminium from 0 to 100 per cent the author prepared a series of nine specimens which were examined under the microscope, the surfaces being attacked by suitable reagents.

Alloys of Strontium with Zinc and Cadmium.—Henri Gautier.—The author first repeats, with strontium chloride, the experiments described by Caron for the preparation of a zinc-calcium alloy from calcium chloride. Under these conditions an alloy of zinc and strontium is formed, but by varying the proportions of the materials employed and the temperature of the reaction, it was impossible to obtain an alloy containing more than 2 to 3 per cent of strontium. To facilitate the liberation of the strontium, and the formation of its alloy with zinc, he treated the strontium chloride with sodium in presence, not of zinc, but of zinc chloride. By this means an alloy containing 12 to 14 per cent of strontium was obtained. To prepare still richer alloys the strontium chloride was replaced by the iodide. Analogous experiments were then made by replacing the zinc by cadmium, a much more volatile metal. The alloys formed were enriched by distillation in a glass vessel at a very low pressure. An 18 to 20 per cent alloy of the alkaline earth metal after three hours' heating became a 28 per cent alloy, and after a whole day's heating a 45 per cent alloy was obtained.

Silicon in the Unstable Ferrosilicides.—P. Lebeau.—The different authors who have investigated the form under which silicon exists in cast iron and steel, have arrived at no definite opinion. The author's present researches on this subject lead him to conclude that the unstable ferrosilicides contain all their silicon in the combined state as $SiFe_2$. This compound being very soluble in an excess of iron easily gives a homogeneous mass on cooling. This substance can only be obtained in the free state from the silicated products when it is present in a greater proportion than that which makes a saturated solution in the iron towards the solidification point. These results are in perfect agreement with M. Le Chatelier's experiments and the micrographic researches of M. Stead.

A New Practical Method of preparing Trichlorated Butylic Alcohol.—Marcel Guédras.—Trichlorated butylic alcohol can be rapidly prepared as follows:—Some caustic potash is placed in a little flask, and upon this, drop by drop with continual shaking, a mixture of chloroform and acetone in equal proportion is poured. During this operation the flask is heated to 30°. When the whole of the mixture has been introduced the flask is kept at a temperature of 50° for an hour. It is then heated to 60°, but must not reach 70°, in order to remove the excess of acetone and chloroform. There then remains in the flask trichlorobutylic alcohol and potash besides impurities. The reaction may be represented by the equation:—

$$CHCl_3+CH_3—CO—CH_3+KOH=$$
$$=CCl_3—\underset{\displaystyle CH_3}{\overset{\displaystyle CH_3}{C}}—OH+KOH.$$

The contents of the flask are treated with a current of steam, which removes the trichlorobutylic alcohol, and which separates out on cooling in the form of white crystals. This alcohol possesses a very characteristic camphorated smell. It melts at 80—81°, and boils at 167°. It is almost insoluble in cold water, but dissolves to the extent of 2 per cent in hot water. It is soluble in ether, benzene, glacial acetic acid, strong alcohol, chloroform, and acetone. The dilute alkalis and acids have no action on it. Trichlorobutylic alcohol is a local anæsthetic, and also possesses antiseptic properties.

MISCELLANEOUS.

British Columbia Institute of Assayers.—At the meeting held at Nelson on November 8th, A. McKillop, chairman, it was unanimously resolved that an Association of the Assayers of British Columbia be forthwith inaugurated. The Constitution and By-laws, as drafted in Committee, were adopted in their entirety by unanimous vote. That this Association should prove of great advantage in many ways to assayers, and indeed to all interested in the Mining industry, is hardly to be doubted. Its value will depend, in a large degree, on the number of certified assayers being included in its membership.—J. Cuthbert Welch, Secretary *pro tem.* Office: Trail, B.C., December 13th, 1901.

MEETINGS FOR THE WEEK.

Monday, 6th.—Society of Chemical Industry, 8. "Report of the Joint Arsenic Committee of the Society of Chemical Industry and of the Society of Public Analysts." "The Retarding Influence of Aldehydes on the Maturation of Spirits," by Prof. J. T. Hewitt, M.A., D.Sc., Ph.D.

Tuesday, 7th. Thursday, 9th. } Royal Institution, 3. (Christmas Lectures to Young People). "On Waves and Ripples in Water, Air, and Æther," by Professor J. A. Fleming, M.A., F.R.S., D.Sc.

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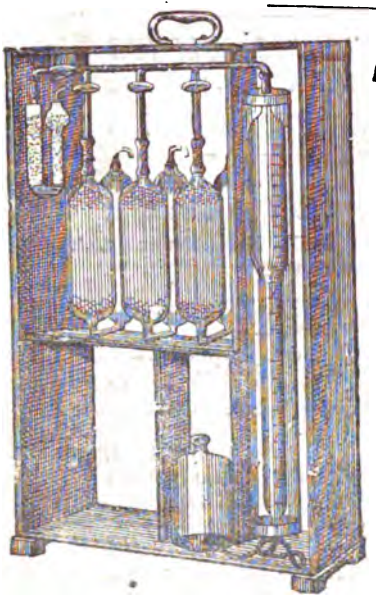

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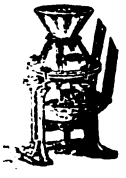

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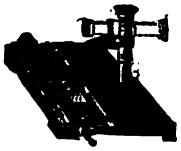

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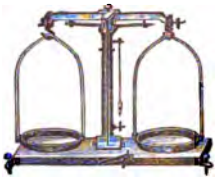

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THE CONSTITUTION OF TOURMALINE.*

By F. W. CLARKE.

SOME years ago, in an extended paper upon the constitution of the silicates (*Bull. U.S. Geol. Survey*, 1895, No. 125), I proposed a formula for tourmaline which seemed to satisfy all known conditions. Recently, Penfield and Foote (*Amer. Journ. Sci.*, 1899, Fourth Series, vol. vii., p. 97) have offered still another interpretation of the analyses, and support their views with a considerable weight of argument. The appearance of their paper has led me to re-scrutinise the evidence more in detail than previously, and the result has been to modify my formulæ in some particulars while retaining them in their general form.

According to Penfield and Foote all tourmaline may be represented as salts of the alumino-borosilicic acid, $H_{11}Al_3B_2Si_4O_{21}$, in which two hydroxyls are permanently linked to boron, leaving an available valency or basicity of nine. In my formulæ all tourmalines are derived from the similar acid $H_{14}Al_5B_3Si_6O_{31}$, with all of the hydrogen atoms replaceable by bases. Upon bringing the two acids to the common basis of six silicon atoms, they compare as follows:—

Penfield and Foote	$H_{16\cdot5}Al_{4\cdot5}B_3Si_6O_{31\cdot5}$
Clarke	$H_{14}Al_5B_3Si_6O_{31}$

Replacing aluminum by hydrogen in order to show the ultimate acids these expressions become:—

Penfield and Foote	$H_{30}B_3Si_6O_{31\cdot5}$
Clarke	$H_{29}B_3Si_6O_{31}$

The small difference between the empirical formulæ is thus made evident, and it hardly amounts to more than the uncertainties in the analyses. In fact, the trustworthy analyses of tourmaline give ratios lying between and beyond both extremes, as the following formulæ, computed from Riggs's data, show. In these expressions fluorine has been assumed equivalent to hydroxyl:—

Pierrepont, black	$H_{28\cdot87}B_{2\cdot93}Si_6O_{30\cdot83}$
Paris, black	$H_{30\cdot73}B_{2\cdot66}Si_6O_{31\cdot35}$
Stony Point, black	$H_{30\cdot23}B_{3\cdot02}Si_6O_{31\cdot65}$
Auburn, colourless	$H_{29\cdot25}B_{2\cdot77}Si_6O_{30\cdot78}$
Brazil, red	$H_{30\cdot79}B_{2\cdot78}Si_6O_{31\cdot56}$
Gouverneur, brown	$H_{28\cdot36}B_{2\cdot95}Si_6O_{30\cdot61}$
Hamburg, brown	$H_{30\cdot93}B_{3\cdot05}Si_6O_{32\cdot14}$

The two analyses by Penfield and Foote, however, conform sharply to their formula, thus:—

De Kalb, white	$H_{29\cdot85}B_{3\cdot03}Si_6O_{31\cdot48}$
Haddam Neck, green ..	$H_{29\cdot98}B_{3\cdot06}Si_6O_{31\cdot59}$

The Gouverneur and Hamburg tourmalines represent the extreme range of variation; a variation which is too large to be safely set aside as due to analytical errors or to impurities in the material analysed. Some of the formulæ approximate to mine, some to that of Penfield and Foote, and hence it seems probable that neither formula, without some qualification, can safely be taken as final.

In order to be satisfactory, a constitutional formula must fulfil several conditions. First, it must adequately express the composition of the compound in question, covering all of its variations. Second, it must be readily applicable to the full discussion of analyses, so that the different isomorphous salts which are commingled in a mineral species can be separately identified and given reasonable expressions. Finally, it should indicate the relations between a species and the other minerals with which it is allied, or into which it commonly alters. A formula can be fully adopted only when all of these conditions are satisfied. The third condition, which relates to function, is equally important with the other two.

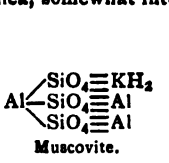
With the tourmalines, the micas seem to be most nearly akin. In each group we have to consider commingling of isomorphous molecules, and when tourmaline alters, a mica is commonly the product of the reaction. In composition, also, the two groups show an apparent parallelism. With the lithia mica, lepidolite, lithia tourmalines occur; with muscovite and biotite, the common iron tourmaline is associated; and the magnesian tourmalines, which show the minimum of alumina in their composition, are similarly allied to phlogopite. This relationship, if it is real, should be suggested in the formulæ assigned to the several species.

To the commoner micas a simple series of formulæ can be easily given thus:—

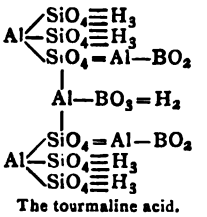
Muscovite	$Al_3(SiO_4)_3KH_2$,
Biotite..	$Al_2(SiO_4)_3Mg_2KH$,
Phlogopite	$Al_2(SiO_4)_3Mg_3KH_2$;

and to these types, or mixtures of them, most micas are referable. The variations and exceptions have been considered elsewhere, and need not be discussed here.

With these fundamental molecules the corresponding salts of the tourmaline acid, $H_{29}B_3Si_6O_{31}$, or $H_{14}Al_5B_3Si_6O_{31}$, are structurally correlated. The subjoined formulæ are sufficient to make this point clear; and to render the splitting up of tourmaline, its alteration into mica, somewhat intelligible.



Muscovite.



The tourmaline acid.

In the acid, two hydrogen atoms are united with the orthoboric group, and twelve with the orthosilicate portion of the nucleus. Hence, to avoid repetition of the structural expression, the formula may be condensed into a linear form as follows:—$Al_5(SiO_4)_6(BO_2)_2.BO_3H_2.H_{12}$, and this is applicable to the discussion of the analyses. For example, Riggs's analysis of the black magnesium tourmaline from Pierrepont, New York, corresponds to the following molecular mixture:—

13. $Al_5(SiO_4)_6(BO_2)_2.BO_3Ca.Mg_4H_4$
7. $Al_5(SiO_4)_6(BO_2)_2.BO_3Mg.Mg_4H_4$
2. $Al_5(SiO_4)_6(BO_2)_2.BO_3Na_2.Al_2Na_4H_2$

Comparing this with the analysis, and reducing the latter by union of like bases and re-calculation to 100 per cent, we have:—

	Found.	Reduced.	Calculated.
SiO_2	35·61	37·19	37·05
B_2O_3	10·15	10·51	10·80
Al_2O_3	25·29	27·10	27·19
Fe_2O_3	0·44		
TiO_2	0·55		
CaO	3·31	3·45	3·40
FeO	8·19	16·31	16·28
MgO	11·07		
Na_2O	1·51	1·72	1·74
K_2O	0·20		
H_2O	3·34	3·72	3·54
F	0·27		
	99·93	100·00	100·00

* *Bulletin of the United States Geological Survey*, No. 167.

The result is evidently satisfactory. In dealing with titanium I have followed Penfield, regarding it as really Ti_2O_3, and equivalent to alumina. The fluorine is treated as replacing hydroxyl, and is therefore united with the water. It is possible, however, that fluorine may sometimes replace the group BO_2, an equivalency which is strongly indicated in the cappelenite group of minerals.

The brown tourmaline from Gouverneur, New York, as analysed by Riggs, also reduces to a similar mixture of molecules, and its composition may be written thus:—

5. $Al_5(SiO_4)_6(BO_2)_2.BO_3Ca.Mg_4H_4$,
3. $Al_5(SiO_4)_6(BO_2)_2.BO_3Mg.Mg_4H_4$,
2. $Al_5(SiO_4)_6(BO_2)_2.BO_3NaH.Al_2Na_2H_4$;

and the comparison between analysis and theory is as follows:—

	Found.	Reduced.	Calculated.
SiO_2	37·39	37·54	37·32
B_2O_3	10·73	10·76	10·88
Al_2O_3	27·79	28·72	28·53
Fe_2O_3	0·10		
TiO_2	1·19		
FeO	0·64	14·51	14·52
MgO	14·09		
CaO	2·78	2·79	2·90
Na_2O	1·72	1·83	1·93
K_2O	0·16		
H_2O	3·83	3·85	3·92
	100·42	100·00	100·00

By consolidating lime with magnesia the expressions for both tourmalines might be simplified; but in other cases this would not be warranted. In some tourmalines calcium seems rather to replace sodium, or else the group NaH, a probability which will appear later.

In these two tourmalines the theoretical silicon-oxygen ratio Si_6O_{31} is assumed, in accordance with my original formula. We may now consider the cases in which that ratio is exceeded, with more or less approach to the formula proposed by Penfield and Foote. This condition is easily satisfied by regarding one of the component salts of tourmaline as slightly basic, containing the bivalent group =Al—O—H or, =Al—F as an essential factor. With this assumption, which recognises the equivalency of hydroxyl and fluorine, the analyses reduce to the general type indicated in the two preceding examples. For instance, the white tourmaline from De Kalb, New York, has the following composition:—

10. $Al_5(SiO_4)_6(BO_2)_2.BO_3(AlOH).Mg_4H_4$.
20. $Al_5(SiO_4)_6(BO_2)_2.BO_3Ca.Mg_4H_4$.
3. $Al_5(SiO_4)_6(BO_2)_2.BO_3Na_2.Al_3Na_3$.

For comparison the analyses by Penfield and Foote and by Riggs are available. In this case the minute quantities of titanium are ignored.

	Found.		Reduced.		Mean.	Calculated.
	P. and F.	Riggs.	P. and F.	Riggs.		
SiO_2 ..	36·72	36·88	36·56	36·81	36·69	36·79
TiO_2 ..	0·05	0·12	—	—	—	—
B_2O_3 ..	10 81	10·58	10·76	10·56	10·66	10·74
Al_2O_3 .	29 68	28·87	29·55	28·81	29·18	29·07
FeO ..	0·22	0·52	14·97	14·79	14·88	14·86
MgO ..	14·92	14·53				
CaO ..	3·49	3·70	3·47	3·69	3·58	3·48
Na_2O .	1·26	1·39	1·28	1·50	1·39	1·44
K_2O ..	0 05	0·18				
H_2O ..	2·98	3·56	3·41	3·84	3·62	3·62
F ..	0·93	0·50				
	101·11	100·83	100·00	100·00	100·00	100·00

The dark brown tourmalines from Orford, New Hampshire, and Monroe, Connecticut, as analysed by Riggs, also reduced to similar form, and approximate to the mixture—

15. $Al_5(SiO_4)_6(BO_2)_2.BO_3(AlOH).Mg_4H_4$,
6. $Al_5(SiO_4)_6(BO_2)_2.BO_3Ca.Mg_4H_4$,
8. $Al_5(SiO_4)_6(BO_2)_2.BO_3NaH.Al_3Na_2H$,

with the comparison as follows:—

	Found.		Reduced.		Calculated
	Orford.	Monroe.	Orford.	Monroe.	
SiO_2 ..	36·66	36·41	36·96	37·34	36·84
B_2O_3 ..	10·07	9·65	10·16	9·89	10·74
Al_2O_3 ..	32·84	31·27	33·28	33·13	33·11
TiO_2 ..	0·23	1·61			
FeO . .	2·50	3·80	11·84	11·88	11·85
MgO ..	10·35	9·47			
CaO ..	1·35	0·98	1·36	1·00	1·19
Na_2O ..	2·42	2·68	2·58	2·88	2·62
K_2O ..	0·22	0·21			
H_2O ..	3·78	3·79	3·82	3·88	3·65
	100·42	99·87	100·00	100·00	100·00

Here the divergence between the composition as found and as calculated is evidently due to the low determinations of boric acid in the analysis. Still the comparison is close.

(To be continued).

THE REACTION BETWEEN CHLORINE AND AMMONIA.*

By WILLIAM A. NOYES and ALBERT C. LYON.

In Hoffmann's well-known lecture experiment for the demonstration of the composition of ammonia, the introduction of the ammonia into the tube filled with chlorine is followed by the addition of dilute sulphuric acid. Some years ago, when performing this experiment, it occurred to one of us that the use of the sulphuric acid was unnecessary, as any excess of ammonia would be absorbed by the large amount of water which is allowed to enter the tube later. The sulphuric acid was accordingly omitted, but with the surprising result that the tube was left only one-sixth full of nitrogen instead of one-third full, as it should have been. Recently an opportunity has been found to give the subject a more careful study.

A glass tube, having a capacity of about 95 c.c., and closed at each end with a stop-cock, was prepared. This tube, after cleaning and drying, was filled with chlorine which was generated from manganese dioxide and hydrochloric acid, and washed with water. The gas was passed through the tube from below till the portion escaping was completely absorbed by a solution of sodium hydroxide. After removing the chlorine from the tubes outside of the stop-cocks, aqua ammonia was allowed to enter the tube from above, and, after shaking gently, was followed by water till the residual gas was at atmospheric pressure. The amount of the nitrogen was then determined by filling the tube with water from a burette which was connected with its lower end.

It was soon found that when strong ammonia is used in excess the volume of the nitrogen is considerably greater than one-sixth of the volume of the chlorine, and may approach one-third of the volume of the chlorine, but never reaches that limit. If, however, a solution containing 0·5 per cent of ammonia is used in such amount as to leave the solution faintly acid, the volume of the nitrogen approaches very closely to one-sixth the volume of the chlorine. If a 2 per cent solution of ammonium chloride is used instead of the ammonia, the chlorine is almost completely absorbed, and the volume of the nitrogen may

* The work described in this paper forms the subject of a thesis by Mr. Lyon for the degree of Bachelor of Science at the Rose Polytechnic Institute. From the *Journal of the American Chemical Society*, xxiii., No. 7.

be as low as only 1 or 2 per cent of the volume of the chlorine. As will be shown below by quantitative results, the normal reaction between chlorine and ammonia is $12NH_3+6Cl_2=N_2+NCl_3+9NH_4Cl$. This normal reaction is obtained, however, only when the ammonia is used very nearly in the proportion indicated. If an excess of ammonia is used, it reacts in part with the nitrogen trichloride, giving free nitrogen, in part, probably, giving ammonium hypochlorite (Schönbein, *Journ. Prakt. Chem.*, lxxxiv., 386; Fresenius, *Zeit. Anal. Chem.*, li., 59).

$$NCl_3+NH_3=N_2+3NH_4Cl;$$
$$NCl_3+2NH_4OH+H_2O=3NH_4ClO.$$

If, on the other hand, too little ammonia is used, the ammonium chloride formed by the reaction acts, in part, on the chlorine as stated above, with little or no evolution of nitrogen, and the volume of nitrogen liberated will be less than one-sixth the volume of the chlorine.

In studing the reaction quantitatively, the tube described above was covered with dark paper to prevent the action of light on the nitrogen trichloride formed. After filling the tube with chlorine an amount of 0·5 per cent ammonia solution, equal to from 90 to 95 per cent of that required by the reaction, was allowed to enter, the tube was shaken gently, and then water of the temperature of the original gas allowed to enter to atmospheric pressure. After gentle shaking a small additional amount of water would enter, indicating that an absorption of some kind had taken place, and the shaking was repeated till no more water would enter. Whether this small final absorption is due to residual chlorine, or to the fact that nitrogen trichloride has an appreciable vapour-pressure, and is not quickly absorbed by the water, was not determined. The lower end of the tube was then connected with a burette and the amount of water required to fill the tube gave the volume of the residual nitrogen.

The contents of the tube were then transferred to a separatory funnel containing 10 c.c. of benzene (Hentschel, *Ber. d. Chem. Ges.*, xxx., 1434), shaken, the benzene separated, and this repeated twice more. The benzene containing the nitrogen trichloride was shaken at once with an excess of a solution of arsenious oxide in sodium bicarbonate, and the excess of arsenious oxide determined with a standard iodine solution. The reaction is $3As_2O_3+2NCl_3+15H_2O=2NH_4Cl+4HCl+6H_3AsO_4$.

Each atom of chlorine present is therefore equivalent to 2 atoms of "available chlorine" (Hentschel, *loc. cit.*).

After titration the solution was distilled with an excess of caustic soda, and the ammonia in the distillate was determined by means of a standard acid, thus proving that the substance extracted by the benzene was really nitrogen trichloride.

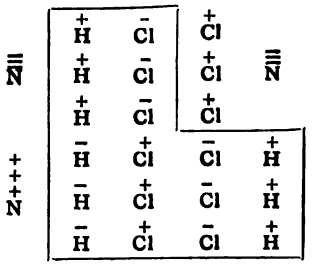
The solution which had been extracted with benzene, and which was, as has been explained, still slightly acid, was titrated with the same ammonia solution first used, and the total ammonia required determined.

The results are as follows:—

	Calculated.	Found. I.	II.	III.	IV.	V.
Volume of nitrogen as compared with chlorine	16 67	14·32	16·31	17·62	17·20	17·05
Available chlorine .	50·0	49·3	43·4	35·7	45·5	41·7
Molecules of ammonia per atom of chlorine	1·0	0·97	1·03	1·04	1·01	0·98
Ammonia from NCl_3. Mols. per mol. of NCl_3 ..	1·0	0·95	1·10	1·10	1·14	—

Owing to the instability of the nitrogen trichloride and to the secondary reactions which can never be entirely avoided, an exact agreement cannot be expected. The results satisfactorily demonstrate that the reaction already given is the primary one between ammonia and chlorine.

Probably the most interesting feature of the reaction here studied is the fact that 6 molecules of chlorine must react *simultaneously* with 3 molecules of ammonia, for, if the liberation of nitrogen by the action of 1 part of the chlorine were independent of the formation of nitrogen trichloride by another part, the constant relation of 1 volume of nitrogen to 6 volumes of chlorine would be improbable. The following hypothesis as to the cause of this relationship is given with some hesitation and in the hope that it may lead to discussion, and to a further consideration of similar cases. If we suppose, what seems not inherently improbable, that all reactions involving the decomposition of molecules are preceded by an ionisation of the parts of those molecules, it would follow that elementary molecules, as well, may ionise into positive and negative parts. This explanation of the reaction may be represented graphically thus:—



The explanation given involves the further idea that in the ionisation of ammonia the hydrogen may become either positive or negative. When we remember the neutral character of ammonia, and the fact that its hydrogen may be replaced by either chlorine or sodium, such a thought is not so improbable as it seems when first presented.

That the hypothesis here suggested is capable of a wide application need hardly be said.

A RAPID METHOD FOR THE DETERMINATION OF ARSENIOUS OXIDE IN PARIS GREEN.

By S. AVERY and H. T. BEANS.

The authors have been working for some time on a method for determining the arsenic in Paris green, in the hope that one might be found that would be both more rapid and accurate than any thus far proposed. As a result we offer the following method, which we believe to be new and which has given most excellent results on a considerable number of samples of Paris green examined in this laboratory.

For the determination, sample the Paris green by quartering (as one would an ore for assaying) down to about 1 grm. Pulverise this small sample in an agate mortar and weigh out 0·2 to 0·3 grm. into a beaker of about 300 c.c. capacity. Add about 25 c.c. of water and to the green suspended in water add, with constant stirring, concentrated hydrochloric acid till solution is just effected; from 6 to 10 drops are usually sufficient. Now add to the acid solution sodium carbonate solution till a slight permanent precipitate is formed, and at this point add 2 to 3 grms. of sodium potassium tartrate in solution. The tartrate will at once dissolve the precipitated copper and prevent further precipitation during the subsequent titration. Dilute to about 200 c.c., add solid sodium bicarbonate and starch solution, and titrate with iodine in the usual way.

The time required for the determination is about ten minutes. The end reaction is sharp and is not in the least obscured by the blue colour of the copper solution.

Triplicate determinations on the same sample of a very

uniform Paris green were as follows, taking 0·3 grm. Paris green for analysis.

Iodine solution c.c.	As_2O_3 Grm.	As_2O_3 Per cent.
34·52	0·17056	56·85
34·59	0·17091	56·97
34·58	0·17086	56·95

To make sure that the presence of copper exerted no influence, several lots of pure arsenious oxide were weighed out. Some of these were titrated as usual and the others were first mixed with about an equal weight of copper sulphate in solution and then with the tartrate according to the method given. No appreciable difference could be observed in the several titrations.

Several other analysts, as well as ourselves, have found that the results, in terms of metallic arsenic, obtained by this method are slightly higher than the results by other methods, even when the latter admit of the determination of arsenic in either stage of oxidation. This fact would seem to indicate that the Paris greens on the market contain arsenic in the lower stage of oxidation only.

Cuprous oxide interferes with the titration, but we have not observed the presence of copper, in this degree of oxidation, in any of the samples examined. It is, of course, possible that adulterants might be added that would affect iodine or iodine salts, but such samples have not as yet been met with, to our knowledge.

In conclusion, we would express our obligations to Dr. H. W. Wiley for his kindness in having the literature of the subject thoroughly searched for our guidance.—*Journal of the American Chemical Society*, xxiii., No. 7.

MODIFIED WILLIAMS METHOD FOR MANGANESE.

By RANDOLPH BOLLING.

THE Williams method, as outlined in Blair's "Chemical Analysis of Iron," although a very accurate one, is unsatisfactory on account of the time required for the filtration of the precipitated manganese dioxide, which, together with a frequently occurring mass of gelatinous silica, chokes up the asbestos filter, and makes the operation more or less tedious.

The following modification has been found to overcome this difficulty entirely, and its adoption has reduced the time of a determination very materially, by eliminating the uncertainty attendant upon the filtration of the slimy manganese dioxide.

Method.

Place 5 grms. of pig-iron drillings in a No. 5 Griffin's beaker, with cover glass, add 75 c.c. nitric acid (sp. gr. 1·20), and after violent action has ceased add approximately 10 c.c. hydrofluoric acid. Give the beaker a circular motion to mix contents, and evaporate down until ferric oxide begins to separate out. Cool, add 100 c.c. nitric acid (sp. gr. 1·42), and heat to boiling. Drop in a spoonful of asbestos fibres, and then add approximately 7 grms. potassium chlorate. Boil until green fumes disappear and nitric acid begins to volatilise. Remove from source of heat, cool rapidly, and filter the manganese dioxide precipitated on the suspended asbestos fibres, on a special filtering tube. Wash twice with strong nitric acid, and rinse out the beaker in which the precipitation was made with cold water, pouring the rinsings through the filter-tube. Continue the rinsing and washing until, when tested with litmus, no acidity is shown. Push the asbestos pad, and precipitate back into the beaker, wash down the sides of the filter-tube to remove all adherent manganese dioxide, and run in 50 c.c. acid ferrous sulphate solution. Disintegrate the asbestos fibres with a stirring rod, and after the precipitate is entirely dissolved, titrate with permanganate. The filtering-tube is prepared by taking a glass tube thin enough to slide loosely down the stem of a carbon funnel, heating one end until it balls, and then pressing out flat. By softening the edge of the flat end, and pressing a nail against the circumference at regular intervals the disk can be made fluted. Slip this rod in a carbon funnel, pushing down until the disk rests solidly, place on it a little dry asbestos, and then pour on it asbestos suspended in water, using a moderate suction to make the pad fairly compact.

Acid Ferrous Sulphate.

Dissolve 20 grms. chemically pure ferrous sulphate in water, add 200 c.c. strong sulphuric acid, and dilute to 2 litres.

Potassium Permanganate Solution.

The strength of this solution should be such that 1 c.c. equals about 0·0056 grm. iron.

Standardisation and Calculation of Results.

To standardise both the ferrous sulphate and the permanganate at the same time, take an iron in which the manganese has been accurately determined, and treat it according to the method given until the precipitate of manganese dioxide is obtained. Dissolve the precipitate in 50 c.c. of ferrous sulphate solution, and titrate with permanganate, then run in 50 c.c. additional of the ferrous sulphate, and titrate again, to obtain the amount of permanganate necessary to oxidise 50 c.c. of the ferrous sulphate solution. By deducting the number of cubic centimetres used in the first titration from the number used in the second, we obtain the volume of solution oxidised by the manganese dioxide. Dividing the percentage of manganese in the sample by the figure thus obtained, and multiplying this by the number of grms. of the original sample, we obtain the value of 1 c.c. permanganate in terms of manganese. For instance, if we take 5 grms. of a pig-iron containing 1·13 per cent manganese and use 4·6 c.c. permanganate in the first titration and 20 8 c.c. in the second titration, then the difference of 16·2 c.c. is the measure of the amount of ferrous sulphate oxidised by the manganese dioxide. Dividing 1·13 by this 16·2, and multiplying this by 5, we obtain the value of 1 c.c., or 0·0034 grm. manganese. The object of standardising in this way, is to carry out the process under conditions similar to those which occur in a regular determination, for the results obtained by titrating clear ferrous sulphate are always lower than when carbon and asbestos are in the solution, for the reason that the eye of the observer notices the rose tint at once in a transparent solution, but the sharpness of the end reaction is slightly obscured by asbestos and carbon, hence the operator runs a little past the true point. So that if both titrations are made under like conditions this error is completely eliminated.

The calculations in a determination are made thus:—If 5 grms. of the sample are taken, 50 c.c. ferrous sulphate equals 20·8 c.c. permanganate; 1 c.c. permanganate equals 0·0034 manganese; 6·2 c.c. are required to oxidise the ferrous sulphate not oxidised by the manganese dioxide.

Then

$$\frac{(20{\cdot}8-6{\cdot}2)\times 0{\cdot}0034\times 100}{5} = \text{per cent manganese.}$$

The time saved by the suspension of the manganese dioxide, and the elimination of silica in the filtering of the manganese dioxide, makes it possible to make a determination in about one hour. The method of standardising solutions is not based on the reaction that is given in the text-books, which is—

$$MnO_2+2FeSO_4+2H_2SO_4=MnSO_4+Fe_2(SO_4)_3+2H_2O,$$

as it was found that solutions standardised on the above reaction were invariably too low in their manganese value. This may be accounted for in the composition of

the precipitate, for it is possible that MnO, Mn_2O_3, Mn_3O_4, Mn_5O_7, may be formed in part. At any rate, if we adopt the MnO_2 theory and standardise accordingly, the method will not give the same result as a sample that has the manganese determined by the basic acetate separation, and the manganese weighed as manganese pyrophosphate according to Gibbs. On this account it is preferable to use a standardised sample of pig-iron.

Below are given some determinations on ores and on some very carefully standardised pig-iron.

Results in the first column are by using the Gibbs method.

In the second column by modified Williams method.

In the third by old Williams method, using the MnO_2 theory.

	Sample No.	Percentage of manganese.		
Iron ores	1.	1·38	1·39	1·11
,,	2.	1·84	1·84	1·51
,,	3.	1·08	1·06	0·91
,,	4.	1·34	1·35	1·20
,,	5.	0·10	0 12	0·08
,,	6.	2·87	2·87	2·14
,,	7.	2·07	2·07	1·83
Pig-iron	8.	0·415	0·41	0·37
,,	9.	0·442	0·44	0·38
,,	10.	0·97	0·99	0·81

In working on ores carrying large amounts of silica it is advisable to dissolve the sample in hydrochloric acid, and evaporate down to dryness, then moisten with hydrochloric acid, and heat until the mass softens, and then add a few cubic centimetres of strong nitric acid; evaporate again until chlorine is driven out, and then add hot water, and filter off the silica, and evaporate filtrate and determine manganese as usual.

I wish to acknowledge my thanks to Mr. W. Walley Davis, chief chemist, for his suggestions in regard to arranging the analytical data for publication.—*Journal of the American Chemical Society*, xxiii., No. 7.

NOTES ON THE ANALYSIS OF EXPLOSIVES.

By F. W. SMITH.

Determination of Sulphur.

In gelatin dynamite containing from 1 to 3 per cent of sulphur, the following method is useful:—Weigh out about 2 grms. into a silver crucible of 100 c.c. capacity; fill it two-thirds full with an alcoholic solution of caustic soda. Warm carefully on a water-bath until the nitroglycerin is decomposed and then evaporate to dryness. Add next about 40 grms. of solid caustic potash and 5 grms. of potassium nitrate. Fuse the contents of the crucible carefully over a blast-lamp until all organic matter is oxidised. Dissolve in dilute acetic acid, filter from a small amount of insoluble matter, and precipitate the sulphates with the usual precautions. The results check very closely.

An Indirect Method for the Estimation of Nitroglycerin in Gelatin Dynamite, &c.

About 15 grms. of the sample are completely extracted with chloroform in a Soxhlet apparatus and the loss in weight noted. In another portion the moisture is determined by desiccation over sulphuric acid for five days, careful experiments having shown that nitroglycerin is not appreciably volatile in a desiccator. Another portion of about 2 grms. is carefully extracted with pure ether by maceration in a small beaker. The ether is poured through a filter and the extraction repeated three or four times. The filtrate is now allowed to evaporate spontaneously or with the aid of a gentle current of air. When the ether is evaporated add about 5 c.c. of ammonium sulphide solution and 10 c.c. of alcohol. Warm gently on a water-bath until the nitroglycerin is decomposed, and then add about 250 c.c. of water and enough hydrochloric acid to give a strongly acid reaction, filter, and wash the precipitate free from acid. Now wash the precipitate out with strong alcohol and chloroform, collecting this filtrate in a weighed platinum dish. Evaporate at a low temperature and dry to constant weight at 50° C. The contents of the dish are now transferred to a silver crucible and the sulphur determined as above described. The increase in weight of the dish less this amount of sulphur represents the chlorform-soluble substances in the original sample except the nitroglycerin, moisture, and sulphur. The percentage of the former substances plus the moisture and sulphur in the original sample gives, when subtracted from the total chloroform-soluble, the percentage of nitroglycerin.

In another portion, the residue in the platinum dish may be investigated for resins, paraffin, &c.

On the Use of Lunge's Nitrometer.

All the glass stop-cocks should be tight. In reference to the practical testing of stop-cocks, see *Journ. Am. Chem. Soc.*, xxi., 430.

Standardising the Nitrometer.—There are two methods applicable, the first of which I shall call "the absolute method," and the second the "empirical method." The latter was devised by Dr. Clarence Quinan, of San Francisco.

The first consists in admitting to the reduction tube a quantity of air such that if reduced to 0° C. and 760 m.m. pressure, it would occupy a volume of 100 c.c. The analysis is then made by weighing out a convenient quantity of nitrate, decomposing it in the usual way, introducing the nitric oxide into the measuring tube, and regulating the pressure so as to bring the volume of air in the reduction tube to 100 c.c., while the surface of the mercury in the measuring tube is at the same level as the surface of the mercury in the reduction tube. If deemed preferable the level of the mercury in the measuring tube may be made to coincide with the level of the mercury in the reservoir tube, so as to make the pressure in the measuring tube equal to the atmospheric pressure. The volume of nitric oxide is then read off and reduced to normal conditions by the usual calculations.

The correctness of the results obtained in this method, aside from errors of manipulation, is affected by the following factors:—

A. The accuracy of the barometer.
B. The accuracy of the thermometer.
C. The accuracy of the graduation of the reduction tube.
D. The accuracy of the graduation of the measuring tube.
E. The accuracy of the weights employed.

It is evident that the work of calibrating all the instruments employed would be very considerable, and it is not safe to assume the correctness of such instruments as they occur in trade.

The empirical method is as follows:—

A sample of potassium nitrate is purified until it shows the absence of impurities by the ordinary tests. It is then dried and the nitric oxide derived from a known quantity is passed into a measuring tube. The quantity of air in the reduction tube is then varied until the volume of the nitric oxide is approximately that calculated from the amount of nitrate taken. The reduction tube is now sealed and a series of analyses of potassium nitrate made with slight variations in the amount taken, and in each case a correction factor determined which shows 100 per cent in purity in the nitrate taken (see example below). The extreme variations in the determinations should not exceed 0·05 per cent. From this series of determinations a correction factor is calculated which is applied to all determinations,

Example.

0·5078 grm. KNO_3 gave 111·7 c.c. nitric oxide.

Log. 111·7	0·04805
Log. 0·5078	0·70569
Log. of c.c. per grm.	0·34236

0·5057 grm. KNO_3 gave 111·2 c.c. nitric oxide.

Log. c c.	0·04610
Log. s.	0·70389
	0·34221

0·5058 grm. KNO_3 gave 111·2 c.c. nitric oxide.

Log. c.c.	0·04610
Log. s.	0·70398
	0·34212

The average of these logarithms gives: Log c.c. per grm. 0·34223 corresponding to 219·9. The correction is obtained by the proportion 221·0 : 219·9, the first term being the theoretical c.c. per grm. for potassium nitrate.

Log. 221·0	0·34428
Log. 219 9	0·34223
	0·00205

In other words, in any analysis the mantissa of the logarithm representing the c.c. per grm. or the percentage of nitrogen must be increased by 0·00205.

Example.

0·5913 grm. gun-cotton gave 119·1 c.c. of nitric oxide.

Log. c.c.	0·07591
Log. s...	0·77181
	0 30410
	0·00205
	0·30615

This corresponds to 202·3 c.c. per grm. or 12·71 per cent nitrogen.

The advantage of this method lies in the fact that all the sources of constant errors A to E are eliminated, no thermometer nor barometer being needed. The weights may be inexact if only consistent. The graduation of the tubes may be inexact and inconsistent. In fact they could be grossly inaccurate without affecting the accuracy of the results if separate corrections were made for each point on the measuring tubes.

The obvious objection to the method lies in the difficulty of preparing a pure potassium nitrate which shall not have a low content of nitrogen by reason of the presence of foreign matter, nor a high content of nitrogen from the presence of sodium nitrate. These objections may be met by preparing samples of potassium nitrate from different sources. All should show the same percentage of nitrogen. Secondly, a sample of nitrate of soda should be purified and the correction factor determined from it independently. Within the limits of experimental error it should be the same as the correction factor derived from potassium nitrate.

Example.

0·4302 grm. sodium nitrate gave 112·5 c.c. nitric oxide.

Log. c.c.	0·05115
Log. s...	0 63367
	0·41748
Log. theoretical c.c. per grm. for sodium nitrate	0·41954
	0·00206

As a third check a sample of pure dry nitroglycerin may be used.

At one time in my work the reduction tube was set by aid of a barometer, since shown to be faulty. A sample of potassium nitrate carefully purified showed in this case about 99 per cent purity, and a sample of sodium nitrate also carefully purified showed the same percentage of purity within the limits of error. The barometer setting of the reduction tube was therefore discarded and the correction factor introduced, since it was manifest that, while either of the nitrates might be impure even after the most careful purification, it was in high degree improbable that both should be impure and of exactly the same degree of impurity.

During the hot weather of summer there is a distinct advantage in having a large subtractive correction factor. In this way the gas may be measured nearly at the pressure of the atmosphere, thus avoiding the considerable strain on the stop-cock and consequent tendency to leakage.

The nitric oxide may be measured either dry or moist, more conveniently, however, in the latter condition. If so measured, a few drops of water should be left in each tube. As the tension of the aqueous vapour is the same in each tube, it may be neglected in the calculations.

The sulphuric acid used in nitrometer work should be free from oxides of nitrogen and iron. It should be of 94 to 95 per cent strength. In acid of 98 per cent strength nitric oxide is quite freely soluble., *Journal of the American Chemical Society*, xxiii., No. 8.

THE VOLUMETRIC DETERMINATION OF ZINC.

By PERCY H. WALKER.

THE most commonly used method for the determination of zinc is the volumetric process of titrating with standard potassium ferrocyanide, using either a uranium solution or one of cobalt nitrate or platinum chloride as indicator. The ferrocyanide titration has several disadvantages. The standard solution does not keep well, and hence must be frequently standardised. If too much ferrocyanide is added in titrating, there is nothing to do but make another determination. The method of using an indicator, by taking out drops, invariably introduces an error.

R. K. Meade (*Journ. Amer. Chem. Soc.*, 1900, xxii., 353) has given us a method based on an entirely different reaction. He precipitates the zinc as zinc ammonium arsenate, and uses this arsenate to liberate iodine, which is then titrated by thiosulphate.

The most satisfactory gravimetric process for determining zinc is the precipitation of zinc ammonium phosphate, and weighing as pyrophosphate, an analogous process being applied to manganese and magnesium.

Many years ago, Stolba (*Chem. Centralbl.* 1866, 727, 728; Sutton's "Volumetric Analysis," Sixth Edition, p. 87) worked out an alkalimetric method for the determination of magnesium, by titrating the magnesium ammonium phosphate with standard acid. Handy's method (*Journ. Amer. Chem. Soc.*, 1900, xxii., 31) is a modification of Stolba's. This process may be much more easily used for the determination of zinc than for magnesium.

The process is carried out as follows:—To the zinc solution, which should also contain ammonium chloride, a large excess of ammonia is added, then a large excess of sodium phosphate The solution remains clear; but if the excess of ammonia is cautiously neutralised, a white cloud is formed as each drop of acid falls into the strong ammoniacal liquid. On stirring this cloud dissolves until nearly all the ammonia is neutralised, when the whole solution becomes milky. It should now be heated to about 75° C., and stirred constantly, at the same time continuing the addition of dilute acid, drop by drop. In a very few minutes the precipitate becomes crystalline, and with care the liquid may be almost perfectly neutralised. It is a good plan to add a small piece of litmus-paper to the liquid; this should not turn red, but

should remain blue or violet, while the hot liquid should have no odour, or only a very faint odour of ammonia. When the precipitation is made as above, the zinc ammonium phosphate is easily filtered, which may be safely done after five minutes' standing. The precipitate should be washed with cold water until the washings show only a faint trace of chlorides, then the paper with the precipitate returned to the beaker in which the precipitation was made, an excess of standard acid added, a few drops of methyl orange, and the exact point of neutrality determined with standard alkali.

According to the equation—

$$ZnNH_4PO_4 + H_2SO_4 = ZnSO_4 + NH_4H_2PO_4,$$

we see that 1 c.c. of normal acid corresponds to 32·7 m.g. zinc. A solution of pure zinc oxide in hydrochloric acid was prepared for testing this method, the following results being obtained:—

	Zinc taken. Grm.	Zinc found. Grm.
1	0·1490	0·1486
2	0·1490	0·1481
3	0 1490	0·1486

Since the zinc ammonium phosphate is not precipitated in presence of a large excess of ammonia, the process may be used in the presence of magnesium which is precipitated in the strongly alkaline liquid, and the filtrate from the precipitate neutralised to precipitate the zinc. About 1·15 grms. of crystallised magnesium sulphate were added to some of the zinc solution, made strongly alkaline with ammonia, sodium phosphate added, and after standing about fifteen minutes with frequent stirring, filtered, washed, and the zinc determined in the filtrate.

Zinc taken	0·1490
Zinc found	0·1481

The process gives fairly good results in the presence of iron, calcium, and magnesium, as the following results will show. Where unknown but rather large quantities of solutions of iron (ferric), calcium, and magnesium salts were added to the zinc solution, the whole being strongly alkaline, sodium phosphate was added in large excess, the solution being in a graduated flask which was filled to the mark, mixed, filtered through dry paper, and an aliquot part taken for determining the zinc.

Zinc taken	0·1192 grm.	0·1192 grm.
Zinc found	0·1172 „	0·1172 „

Manganese, however, must be previously separated, best by the nitric acid and potassium chlorate method.—*Journal of the American Chemical Society*, xxiii., No. 7.

PRELIMINARY NOTE ON THE PREPARATION OF BARIUM.*

By EDGAR STANSFIELD, B.Sc.

Many attempts have been made to prepare metallic barium, but, up to the present, no satisfactory method of obtaining it seems to have been described. There is a general lack of chemical analyses of the products obtained by the various workers, even when they profess to have obtained the metal.

In view of the satisfactory solution by Moissan (*Ann. Chim. Phys.*, 1899, [7], xviii., p. 289), of the difficulties of preparing the allied metal calcium, which can now be obtained in a pure state, I decided to undertake a critical study of the hitherto proposed methods of preparing barium, in the hope of finding some modified or new process which would give good results; care being taken to check each step by as accurate a chemical analysis as possible.

At present I propose to consider three of these:—

1. *Method of Distillation of Amalgam.*—My results are here in accordance with those of the more modern experimenters (Kern, *Zeits. Anorg. Chem.*, 1898, xvii., p. 284; also 1900, xxv., p. 1; Langbein, "Inaugural Dissertation," Königsberg, 1900). I found that, although it is easy to prepare a crystalline amalgam, containing nearly 5 per cent of barium, by means of the electrolysis of a saturated solution of barium chloride, using a mercury cathode, I was unable to remove all the mercury from this amalgam by distillation *in vacuo*. It is very difficult to completely dry the amalgam without oxidation occurring.

2. *Method of Preparing Zinc Alloys.*—I have tried two methods for this preparation. The first, which consists in electrolysing a fused mixture of barium and sodium chlorides with a molten zinc cathode, was wholly unsuccessful; the second, which is the method used by Caron (*Comptes Rendus*, xlviii.. 440), yielded better results. In the best experiments I took 210 grms. zinc, 20 grms. sodium, 120 grms. barium chloride (20 grms. sodium being equivalent to 90 grms. barium chloride); I also added 25 grms. of sodium chloride as a flux. This mixture was heated in a fire-clay crucible until it boiled, and was then allowed to cool. A porous metallic mass was obtained weighing 250 grms., analysis showing that this contained 12 per cent of barium, as well as traces of chlorides and oxides; but that at least some of the barium was present in the metallic state was shown by the freedom with which it decomposed water, especially after it had been powdered.

This product did not seem pure enough to be likely to give good results on distilling off the zinc. The disadvantage of the method of distilling off the mercury or zinc from the amalgam or alloy of barium, seems to be that, at the best, only a product of finely divided material can be obtained; such powdery masses are of very indefinite composition, and, presenting, as they do, so much surface to the action of the surrounding medium, offer conisiderable difficulty to the working up of a satisfactory product from them.

3. "*The Goldschmidt Process.*"—It appeared therefore advisable to find some method which would give a fused product, such product being likely to have a more homogeneous composition. Since barium evidently has a rather high melting-point, such a process must be one carried out at a high temperature. The Goldschmidt process, whereby oxides are reduced by means of finely divided aluminium mixed with them in an intimate manner, and in which the heat of the reaction, being very large, is generally sufficient to fuse even the alumina formed, was considered likely to give interesting results.

The experiments had to be carried out under reduced pressure, both in order that the charge should not be scattered about by the sudden expansion of the air, due to the rise of temperature, and to prevent loss owing to the formation of barium oxide and nitride, the ease with which these compounds are formed being a great hindrance to all attempts to prepare the metal in the atmosphere.

Description of Apparatus.—Crucibles of Veitsch magnesite of about 200 c.c. capacity were employed; these do not seem to be acted on during the experiment. One crucible generally lasted for two or three experiments before it broke up owing to the sudden changes of heat. Lids were made out of powdered Veitsch magnesite with magnesium chloride solution. The crucible was strengthened by being surrounded by a sheet-iron jacket, and the lid was held down on to the crucible by two brass plates, one below the crucible and the other on the top of the lid, these plates being tightly screwed together by means of three bolts. The crucible, thus encased, was placed inside an iron vessel, over which another iron vessel, inverted, was placed, this being a precaution for the safety of the bell-jar desiccator, inside which the whole apparatus was finally put. Undue heating of the

* Read before the Manchester Literary and Philosophical Society, October 29, 1901. From the *Memoirs and Proceedings of the Manchester Literary and Philosophical Society*, xlvi., Part 1.

iron vessels by conduction was guarded against by means of asbestos millboard.

The charge is fired by the following contrivance:—Through the sides of the crucible, and near the bottom, were drilled two holes opposite to each other; two aluminium wires were passed through these and joined inside by a bridge of fine platinum wire, which passed through the centre of the starting charge. The aluminium wires were connected by binding screws to two lengths of insulated copper wire, which passed air-tight through the stopper of the desiccator. To fire the charge, an electric current of 2 to 3 ampères was passed through the wires, the platinum wire becoming hot enough to start the reaction.

In the earlier experiments the charge consisted of barium oxide and aluminium, together with a starting charge of barium peroxide and aluminium. It was afterwards found to be more satisfactory to use nothing but the peroxide and aluminium; for not only can the peroxide be obtained in a purer condition, but, the heat of the reaction being greater, a better-fused mass is obtained. The aluminium used was in fine granules, except round the platinum wire, where the flake aluminium was used; it is necessary to strongly heat both sorts before use in order to drive off the fatty matter with which they are always contaminated.

In an actual experiment, all the chemicals to be used and the crucible are strongly heated, and allowed to cool in a desiccator, the charge is then weighed out, and the apparatus fitted up as rapidly as possible to prevent absorption of moisture. The bell-jar desiccator containing the apparatus is then exhausted by means of a water pump, and the charge fired.

After the apparatus has cooled, there remains a grey mass at the bottom of the crucible, which usually contains crystalline flakes of a silver-white metal.

Both the grey mass and the metallic particles decompose water very rapidly. In dry air they do not change, but in moist air they rapidly crumble up, forming a white powder, while if heated in a blowpipe flame they first scintillate slightly, but then becoming coated with oxide they suffer no further action, nor are there any signs of fusion.

In the best experiment, the charge consisted of 100 grms. of barium peroxide, 21 grms. of aluminium (these being in the proportion required by the equation $3BaO_2+4Al=3Ba+2Al_2O_3$), together with 25 grms. of the product of a previous experiment. The product in this experiment was a very hard, almost black mass, which, however, was in parts very rich in crystalline flakes of metal, and also contained some small nodules of metal.

The metallic part contained 63·3 per cent barium, but only 19·3 per cent aluminium, the total being 82·6 per cent. An analysis in another experiment showed 66·6 per cent barium, 29·3 per cent aluminium; total 95·9 per cent.

The analysis of the nodules mentioned above, whilst it gave but 58 per cent barium, was the only case in which the full 100 per cent of barium + aluminium was reached; usually it was impossible to completely separate the flakes of metal from the associated oxide for the purpose of analysis, as the metal was so very brittle.

These experiments seem to indicate that the reaction is reversible, and that it cannot be used to obtain pure barium, but only an alloy of barium with aluminium containing up to about 60 per cent barium.

An endeavour was made to prepare a calcium alloy by this method, but the reaction would not proceed at all. It was also attempted to replace the aluminium by magnesium; in this experiment, the charge taken was only 14 grms. magnesium and 48 grms. barium peroxide, but the reaction was violent and shattered the bell-jar.

I hope to study other methods of preparation, more particularly the electrolysis of barium compounds.

The above work was carried out in the Electro-Chemical Laboratory of the Owens College.

ON TRIVALENT CARBON.

(Third Paper).*

By M. GOMBERG.

It has been shown in the papers published by me on this subject that by the action of metals upon triphenylchlormethane the halogen can be removed quantitatively, and that there results an extremely unsaturated hydrocarbon. From the behaviour of this substance towards halogens and towards oxygen, the conclusion was drawn that it is an unsaturated radicle, triphenylmethyl. It was shown that the same body results whether carbon disulphide, benzene, ether, or acetic ether is employed as a solvent. It was stated that in the case of the first two mentioned solvents the unsaturated hydrocarbon remains in solution, while with ether or acetic ester as a solvent the hydrocarbon separates in the form of large transparent crystals. This crystalline body possessed all the properties of the unsaturated hydrocarbon, and was also identical with the substance which was obtained when triphenylchlormethane in benzene was acted upon by zinc and the resulting hydrocarbon was precipitated from its concentrated solution by means of ether or acetic ester. Facts have since been discovered which necessitate a modification of this view, and I desire to publish the results even in their present incomplete state.

The Action of Zinc in Ether as a Solvent.

When triphenylchlormethane is dissolved in absolute ether and a strip of zinc is introduced, the solution at once turns yellow and a dark viscous mass begins to separate. After a while crystals begin to form which adhere to the zinc and to the sides of the vessel. If the reaction be carried on in a sealed tube it will be noticed that in a few days the black mass begins to diminish in quantity, and in about three weeks it will disappear completely. The solution still remains yellow, but the crystals are colourless. It is impossible to get the ether and the reagents so absolutely free from moisture but that at least a few bubbles of hydrogen will be formed during the reaction (*Am. Chem. Journ.*, xxv., 320). The viscous mass which is formed at the beginning of the reaction is not zinc chloride, but a double salt of it with triphenylchlormethane (*Journ. Am. Chem. Soc.*, xxii., 761). Zinc chloride, I find, is soluble in ether, while the double salt is not. The thick yellow mass which was described under the experiments with benzene is also a double salt of a similar or of identical composition, and not a compound of zinc chloride with benzene, as has been previously supposed. Such a double salt when treated with water would give up all its chlorine to the latter, and this accounts for the somewhat misleading results which were obtained in the estimation of chlorine in the insoluble residue from benzene (*Journ. Am. Chem. Soc.*, xxii., 761). On allowing the ethereal solution to stand for some weeks the double salt is completely broken up by the metallic zinc, but in benzene this does not occur. There is no separation of the double salt when acetic ether is employed as a solvent, for the simple reason that the salt is very soluble in this solvent.

The same double salt is formed with the greatest readiness when a solution of zinc chloride in ether or in acetic ether is added to a solution of triphenylchlormethane in benzene or in absolute ether. All attempts to bring it to crystallisation have failed, but a number of other double salts of triphenylhalogenmethanes, and especially of tritolylchlormethane, were obtained in sufficient purity to establish the remarkable tendency of these halogen bodies towards the formation of double compounds with metallic salts. Substances of the following composition

* From the *Journ. Am. Chem. Soc.*, xxiii., No. 7. The first paper appeared in the *Journ. Am. Chem. Soc.*, xxii., 757; the second in *Am. Chem. Journ.*, xxv., 320.

were obtained, and they will be fully described in a later paper :—

$2(C_6H_5)_3CCl.3HgCl_2$, $(C_6H_5)_3CBr.3HgBr_2$, $(C_6H_4CH_3)_3CCl.ZnCl_2$, $(C_6H_4CH_3)_3CCl.HgCl_2$, $(C_6H_4CH_3)_3CCl.FeCl_3$.

All these compounds are beautifully crystalline, and are characterised by intense colouration, from yellow to red. There is no doubt that the yellow colour which is imparted to the solution when zinc or any metal acts upon triphenylchlormethane is due in a large measure, but not entirely, to the formation of these double salts. The transparent yellow crystals which are formed when a solution of triphenylchlormethane in ether or in acetic ether is treated with zinc are not triphenylmethyl itself, but a compound of that with each of the two solvents respectively.

Compound of the Unsaturated Hydrocarbon with Ether.

Samples of the above-mentioned yellow crystalline compound were analysed from time to time, but the results usually fell from 2 to 3 per cent short of 100 for the sum of carbon and hydrogen. This was at first ascribed to the absorption of oxygen by the unsaturated hydrocarbon during the washing and transference of the material. I then came back to the use of benzene as a solvent. An apparatus was constructed by means of which the benzene solution of the hydrocarbon could be concentrated in a vacuum at 30° C.; ether could then be added, and the crystals that were so formed could be filtered, washed, and thoroughly dried in an atmosphere of carbon dioxide —all these operations being conducted in the same single piece of apparatus, without any rubber connections whatever. The crystalline product obtained in this way is almost insoluble in ether. It was washed with ether from 15 to 20 times to insure the complete removal of the accompanying products, such as triphenylmethane, triphenylcarbinol, &c. The crystals are at first perfectly colourless, but soon turn pale yellow, the colour increasing with standing. Twenty grms. of triphenylchlormethane give about 7 grms. of the insoluble crystalline compound. Molecular weight determinations of this substance were first made and the results were fairly satisfactory as calculated for triphenylmethyl.* An elementary analysis, however, gave the following results :—

0·2884 grm. substance gave 0·9488 grm. carbon dioxide and 0·1900 grm. water.

	Calculated for $(C_6H_5)_3C$.	Found.
Carbon	93·76	89·72
Hydrogen	6·24	7·37

A fresh sample was then prepared in a similar way and was dried over sulphuric acid in a vacuum desiccator (previously filled with carbon dioxide) over night. It gave the following results :—

0·2375 grm. substance gave 0·7832 grm. carbon dioxide and 0·1560 grm. water.

	Per cent.
Carbon	89·94
Hydrogen	7·35

A new sample was then prepared, dried, and analysed in the same day, but the results were almost the same.

0·2447 grm. substance gave 0·8060 grm. carbon dioxide and 0·1585 grm. water.

	Per cent.
Carbon	89·83
Hydrogen	7·25

The next sample was prepared by the addition of a mixture of acetic ether and acetic acid instead of absolute ether, in order to hold better in solution any zinc salts which might possibly have accompanied the previously mentioned samples. This sample was washed first with acetic ether and then with absolute ether. It looked whiter and more distinctly crystalline than any before obtained, but the analysis gave figures for carbon and hydrogen the sum of which was only 95·67 per cent. The close concordance of the first three samples, and the fact that when acetic ether, which carries almost twice as much oxygen as ethyl ether, is used for precipitating the hydrocarbon, a product proportionately richer in oxygen is obtained, suggested the idea that the substances analysed must be compounds of the hydrocarbon with the solvents.

	Calculated for $2(C_6H_5)_3C+(C_2H_5)_2O$	Found. I.	II.	III.
Carbon ..	89·93	89·72	89·94	89·83
Hydrogen	7·21	7·37	7·35	7·25

That we have here such a compound is proved by the following experiment :—0·9430 grm. of the ether compound was placed in a platinum boat, and gently heated at 70° C. in a tube, in a stream of carbon dioxide. The loss in weight was 0·1090 grm.

	Calculated for $2(C_6H_5)_3C+(C_2H_5)_2O$.	Found.
Loss	13·22	11·56

The escaping ether was easily identified by its odour, and a few droplets were also condensed in a small U-tube which was sealed on to the further end of the tube. The amorphous yellow residue shows all the properties of the unsaturated hydrocarbon. It absorbs iodine with great eagerness; a solution of it, when exposed to air, gives the triphenylmethyl peroxide. An analysis of the residue gave the following results :—

0·1620 grm. substance gave 0·5520 grm. carbon dioxide and 0·1020 grm. water.

	Calculated for $(C_6H_5)_3C$.	Found.
Carbon	93 76	92·93
Hydrogen..	6 24	7·05

It remains yet to be settled whether this residue represents pure triphenylmethyl or a mixture of it with some of its decomposition products.

(To be continued).

* The determinations were made by the method of the lowering of the freezing-point, in an atmosphere of nitrogen, as carbon dioxide was found to be soluble in benzene to a sufficient degree to impair the results.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, December 19th, 1901.

Prof. J. Emerson Reynolds, F.R.S., President, in the Chair.

(Concluded from p. 10).

178. "*The Influence of Salts and other Substances on the Vapour Pressure of Aqueous Ammonia Solution.*" By E. P. Perman.

The author has investigated the effect produced by urea, mannite, potassium sulphate, ammonium chloride, and copper sulphate respectively on the vapour pressure of aqueous ammonia solution by a method similar to that described for sodium sulphate (*Trans.*, 1901, lxxix., 725). The objects of the research were to find (1) the effect upon the pressure of substances which have no direct chemical action on the ammonia, (2) the effect of rise of temperature on copper sulphate ammonia solution, (3) evidence for or against the existence of hydrates in solution.

The conclusions arrived at by the author are:—(1) That salts of the alkalis have a great effect in raising the pressure, but that the effect of other substances which might be expected to have no direct chemical action on the ammonia is either small or nothing.

(2). That copper sulphate forms complexes with

ammonia in solution which tend to decompose on heating, especially when the proportion of copper sulphate is small.

(3). The effect of potassium sulphate is similar to that of sodium sulphate in raising the pressure. There is but little reason to suspect the existence of a hydrate of potassium sulphate, as it crystallises without water; consequently it would appear that neither sulphate exists in solution as a hydrate.

179. "*The Action of Sodium Hypochlorite on Benzenesulphonanilide.*" (Preliminary Notice). By J. B. Cohen and J. T. Thompson.

In the preparation of the different isomeric dichlorotoluenes (Cohen and Dakin, *Trans.*, 1901, lxxix., 1111), the authors had frequent recourse to the method of chlorination recently studied by Chattaway and Orton (*Trans.*, 1899, lxxv., 1046; 1900, lxxvii., 134 and 789, and *Ber.*, 1899, xxxii., 3573). The authors have found that a very similar reaction occurs with the aromatic sulphonanilides, of which, with Dr. Chattaway's friendly acquiescence, the authors now give a brief notice. Twenty grms. of benzenesulphonanilide were dissolved in 200 c.c. of a solution of sodium hypochlorite (1 c.c. = 0·03 grm. Cl) in the cold and allowed to stand twelve hours. The brown solution was acidified with acetic acid until the buff precipitate re-dissolved, when it was extracted with chloroform. After rapidly evaporating off the solvent, the residual red liquid was digested for an hour with two volumes of glacial acetic acid containing a few drops of concentrated sulphuric acid, until, on pouring into water, a solid substance separated. The new compound was re-crystallised from acetic acid and then from alcohol, when it melted at 129—130°. The yield amounted to 18 grms. of crude or 11 grms. of purified substance.

The mother-liquor contained a small quantity of a semi-solid substance, which became crystalline on standing and after re-crystallisation from benzene and petroleum melted at 116°. This substance is probably the benzenesulphonyl-*p*-chloranilide (m.p. 121°), but it is difficult to free it from oily impurity. The substance melting at 129—130° was analysed, with the following result:—

0·292 gave 14·15 c.c. moist nitrogen at 15·5° and 758 m.m. N = 5·66.
0·3185 ,, 0·1764 AgCl. Cl = 13·57.
0·2097 ,, 0·1820 $BaSO_4$. S = 11·93.
$C_{12}H_{10}O_2NSCl$ requires N = 5·23; Cl = 13·27; S = 11·90.

In order to determine the constitution of the substance, it was hydrolysed. Two grms. were heated with about 8 grms. of concentrated hydrochloric acid in a sealed tube to 190° for four to five hours. On opening the tube, there was a strong smell of benzene, the presence of which was confirmed by extracting with light petroleum, nitrating the extract with a mixture of concentrated sulphuric and nitric acids, washing, and evaporating off the light petroleum. On reducing the residue with zinc dust and acetic acid, aniline was formed, and gave an intense violet colouration with sodium hypochlorite solution. The appearance of benzene in the decomposition of the sulphonanilide compound is readily accounted for, seeing that Armstrong and Field (*Ber.*, 1874, vii., 406) and Jacobsen (*Ber.*, 1876, ix., 258) have shown that sulphonic acids are converted into the hydrocarbons by strong hydrochloric acid under pressure. The acid solution, after extracting with light petroleum, was made alkaline with sodium carbonate, and distilled in steam. The distillate was extracted with ether, the ether removed, and the residue dehydrated in vacuo. A yellow oil weighing 0·8 grm. remained. The substance did not solidify on introducing a crystal of *p* chloraniline, even when cooled in ice; it yielded an acetyl derivative melting at 85—86°, and a benzoyl derivative melting at 102°. This corresponds to *o*-chloraniline, which was prepared from *o*-nitraniline, and then converted into the acetyl and benzoyl derivatives having the above melting-points. In further confirmation, the *o*-chloraniline was heated with benzenesulphonic chloride, and converted into the sulphonanilide melting at 129—130°, which agrees with the melting-point of the product obtained by the action of sodium hypochlorite on benzenesulphonanilide. The reaction, therefore, probably occurs in two steps, as in the chlorination of the acetyl derivatives studied by Chattaway and Orton—

$$C_6H_5SO_2NHC_6H_5 + Cl_2 = C_6H_5SO_2{\cdot}NCl{\cdot}C_6H_5 + HCl,$$
$$C_6H_5SO_2.NCl{\cdot}C_6H_5 = C_6H_5SO_2{\cdot}NH{\cdot}C_6H_4Cl,$$

with the formation mainly of benzenesulphonyl-*o*-chloranilide and a small quantity of the *p*-chloranilide.

Wallach (*Ber.*, 1877, ix., 424) found that benzenesulphonanilide, when heated to 100° with phosphorus pentachloride, yields benzenesulphonyl-*p*-chloranilide, m. p. 121°; but no reference is made to an *o*-compound. The authors have repeated Wallach's work, which they can confirm. The product is difficult to purify, and only a small yield of pure substance could be obtained. From *p*-chloraniline and benzene sulphonic chloride the authors have also synthesised this substance, which melts at 121° as stated by Wallach.

To complete the series, the authors have synthesised the benzene sulphonyl-*m*-chloranilide from *m*-chloraniline, which has not been previously described; like the *p*-compound, it melts at 121°. It gave the following result on analysis:—

0·2576 gave 12·85 c.c. moist nitrogen at 13° and 741 m.m. N = 5·76. $C_{12}H_{10}O_2NSCl$ requires N = 5·23 per cent.

The authors intend to continue the investigation.

180. "*The Relationship between the Substitution and the Constitution of Benzeneazo-α-naphthol.*" By J. T. Hewitt and S. J. M. Auld.

Möhlau and Kegel (*Ber.*, 1900, xxxiii., 2858) have shown that benzeneazo-α-naphthol reacts with Michler's hydrol (tetramethyldiaminobenzhydrol) to form a condensation product in the same manner as paraquinones and their derivatives. Moreover, the substance obtained acetylates in such a manner that the entering acetyl group attaches itself to a nitrogen atom. The authors mentioned draw the conclusion that benzeneazo α-naphthol and its derivatives are of quinone-hydrazone type.

The results obtained by the authors of the present communication are in favour of an oxyazo-formula. Benzeneazo α-naphthyl acetate yields aniline on complete fission and no acetanilde. By partial reduction, a hydrazo-compound is obtained melting about 160—165°; the formula $C_6H_5{\cdot}NH{\cdot}NH{\cdot}C_{10}H_6{\cdot}O{\cdot}COCH_3$ is confirmed by the insolubility of the substance in dilute alkalis.

Nitric acid is not a satisfactory substituting agent to use with benzeneazo-α-naphthol, but bromine reacts with the azonaphthol in presence of acetic acid and sodium acetate, forming a *monobromo* derivative of m. p. 196°. This substance yields aniline on fission; the bromine therefore enters the naphthol nucleus. From this benzeneazobromonaphthol the *ethyl ether*, m. p. 220° (uncorr.), and *acetyl* derivative, m. p. 146°, have been prepared.

The following substances containing bromine in the benzene nucleus have been prepared for purposes of comparison.

o-*Bromobenzeneazo-α-naphthol*, m. p. 183°. o-*Bromobenzeneazo-α-naphthyl acetate*, m. p. 123°. m-*Bromobenzeneazo-α-naphthol*, m. p. 211° (uncorr). m-*Bromobenzeneazo-α-naphthyl acetate*, m. p. 112°. *p*-Bromobenzeneazo-α-naphthol, m. p. 237—238° (Bamberger, *Ber.*, 1895, xxviii., 1896). p-*Bromobenzeneazo-α naphthyl acetate*, m. p. 141°.

The bromination of benzeneazo-β-naphthol is now being studied.

Addendum to Discussion.

On Messrs. Hall and Plyman's paper, No. 162, *on* "*The Determination of Available Plant Food in Soils by the use of Dilute Solvents.*"

Dr. Dyer pointed out that any process must necessarily be empirical. The actions of the root sap of plants could not be exactly imitated, and much must always depend, not only upon the composition of the solution used, but also upon its volume and the duration of contact. He was

very glad that the 1 per cent citric acid solution had on the whole given more reliable indications of the degree of comparative fertility as regarded both phosphoric acid and potash than did the other acid solutions tried. When he was in America a year ago, he found in use for the determination of "available" phosphoric acid a one-fifth normal solution of hydrochloric acid, but this did not give satisfactory results for potash, and he understood that at that time they had not decided in America what was the best solution to use for potash. As to the limits which he had laid down as indicating need of manurial phosphoric acid or potash, both in his original paper on the Hoos Field soils, and in a paper on the Broadbalk soils (*Phil. Trans.*, 1901, clciv. B, 235), he had pointed out that the limits which he gave related to cereal crops, and might probably have to be modified with reference to root and other crops. In this direction, much more work was required. Dr. Dyer called attention to some experiments recently carried out by Mr. Ingle, of the Yorkshire College, Leeds, in which he had extracted a large quantity of soil with 1 per cent citric acid solution, and after washing out had grown various crops in it in pot-culture, with and without total or partial replacement of the constituents removed. Some of Mr. Ingle's photographs were exhibited, which showed that the soil when exhausted of its "available" constituents by 1 per cent citric acid solution, was hardly able to support the life of bean plants, but that when the constituents thus dissolved out were replaced the plants flourished abundantly. Mr. Ingle's experiments were not yet completed, but so far they appeared to confirm the practical value of 1 per cent citric acid solution as a reagent in soil analysis.

Dr. J. A. Voelcker remarked that the experiments of the authors had shown very clearly the influence of one constituent of the soil upon another. Mr. Hall had himself pointed out the variations caused by the presence or absence of lime, and Dr. Dyer had shown how the action of citric acid was influenced by the presence of sodium and magnesium salts, and how irregular were the results when farm-yard manure had been applied to the land. He did not think it was right to take one particular solvent and of one particular strength, and to apply it indiscriminately to all classes of soils alike, nor did he think that the use of hydrochloric acid should be abandoned on the grounds given. That a 1 per cent solution showed, with certain soils, results not consistent with those of certain other solvents or with those of a strong solution of the acid afforded no proof that the acid was an unsuitable solvent. In work on which he had lately been engaged, he had found a 1 per cent citric acid solution useful in many cases, and it was certainly an advance on the older methods, but he was not altogether satisfied with it as yet, and had not found the results to be in all cases consistent with the conclusions arrived at from practical experience of the land. It was not improbable that a distinction would have to be drawn between the two different classes of phosphoric acid present in the soil, the one the phosphoric acid naturally belonging to the soil, the other the phosphoric acid accumulated by repeated manurial applications.

Royal Institution.—On Tuesday next, January 14, Dr. A. Macfadyen, Fullerian Professor of Physiology, R.I., will deliver the first of a course of six lectures on "The Cell, its means of Offence and Defence, Immunity"; on Thursday, January 16, Dr. A. S. Murray will begin a course of three lectures on "Recent Excavations at Delphi and in the Greek Islands"; and on Saturday, January 18, Mr. W. H. Hadow will deliver the first of four lectures (with Musical Illustrations) on "Landmarks in the History of Opera." The Friday evening discourse on January 17, will be delivered by Lord Rayleigh, his subject being "Interference of Sound"; on January 24, Mr. H. G. Wells will give a discourse on "The Discovery of the Future"; and on January 31 Prof. A. Crum Brown one on the "Ions of Electrolysis."

NOTICES OF BOOKS.

The Chemical Analysis of Iron. A Complete Account of all the best known Methods for the Analysis of Iron, Steel, Pig-iron, Iron Ore, Limestone, Slag, Clay, Sand, Coal, Coke, and Furnace and Producer Gases. By Andrew Alexander Blair. Fourth Edition. London and Philadelphia: J. B. Lippincott Company, 1901. Pp. 319.

Owing to the number of improved methods available, the use of new materials in technical work, and the general advance in chemical knowledge, the author found so many changes necessary to prepare the fourth edition of this work, that he decided to re-write it entirely. In doing this he has substituted chemical terms for formulæ in the text, omitted the marginal notes, and changed the nomenclature to coincide with that in general use. These changes will doubtless recommend themselves to the reader as they all tend towards improvement, an aim to be kept in view in new editions of every work.

After describing the necessary apparatus, reagents, &c., the author goes fully into the methods of analysis of pig-iron, bar-iron, and steel, taking seriatim the estimation of the sulphur, silicon, slag and oxides, phosphorus, carbon, titanium, copper, nickel and cobalt, tungsten, and in fact all the metals, both rare and common, which may be met with in iron. A good deal of attention is paid to the estimation of the carbon in its different forms, both graphitic and combined.

Then follow methods for the analysis of ferrochrome, ferrosilicon, and ferrotitanium; iron ores and their analysis come next in order, then the analysis of limestone, clay, slags, coal and coke, &c., and finally the analysis of gases.

The book is very well printed and bound, and besides its high technical value it forms a handsome volume for the laboratory book-shelf. The table of contents and the index are all that can be desired.

Subject List of Works on Chemistry and Chemical Technology in the Library of the Patent Office. London: Darling and Son, Ltd., and The Patent Office. 1901. Pp. 105.

The book now before us is one of a series being issued by the Patent Office. Each volume of the series will consist of two parts: (*a*) a general alphabet of subject headings, with descriptive entries, in chronological order, of the works arranged under these headings; (*b*) a Key or summary of these headings shown in class order. Under each of these headings the searcher will find entries of all works in the Library which are exclusively devoted to the subject of that heading. Works, however, which deal with the subject of a heading in conjunction with other subjects may have to be looked for under a heading of wider scope.

The present list comprises 885 works (79 serials and 806 text-books, &c.), representing some 3300 volumes, and will, without doubt, be found of great service to searchers and other users of the Library.

Introduction to Physical Chemistry. By James Walker, D.Sc., Ph.D., F.R.S. Second Edition. London: Macmillan and Co., Ltd. New York: The Macmillan Co. 1901. Pp. 343.

The short time which has elapsed since the first edition of this book was published in 1891, is good evidence of the favour with which it was received.

Naturally the general aspect of Physical Chemistry has undergone but little, if any, change. The book, however, was never intended as a complete, or even systematic survey of physical chemistry, but more as an explanatory medium. There has always been too great a tendency to keep ordinary chemistry and physical chemistry apart, with the result that the two branches of what may be almost considered the same science have, to a certain extent, interfered with each other in the students' mind,

instead of being mutually helpful. The author's endeavour was to remedy this state of affairs, and, that he has been successful is shown by the increasing demand for his work.

This edition differs from the first one principally in matters of detail, which have been suggested by its use as a text-book.

Among the new matter incorporated in this edition, may be mentioned Daniel Berthelot's method for determining exact molecular weights from the limiting densities of gases, Traube's volume researches, and the position of the recently-discovered atmospheric gases in the periodic system.

The book contains a table of contents and index, and its price is 10s.

Lehrbuch der Chemie und Mineralogie. By Professor G. SIEBERT. Braunschweig: Friedrich Viewig und Sohn. 1901.

IN this book, which is intended for beginners, the whole ground of inorganic and elementary organic chemistry is covered. The first part contains one hundred experiments introductory to the detailed study of inorganic substances, and renders most simple chemical processes familiar to the student, but the arrangement is not altogether good; many of the experiments would have been better deferred to the second part, which gives a systematic account of the inorganic elements and compounds. In most cases the details of the experiments are clearly and fully described, although the accuracy is not always unimpeachable; we notice, for instance, a method given for preparing copper carbonate $CuCO_3$ by precipitation of a copper sulphate solution with sodium carbonate, though this error is corrected in the second volume. The third volume deals with organic chemistry and exhibits no particular features to mark it out from many other books of similar scope; it is doubtful, however, whether the order in which the substances are treated might not be improved upon somewhat; and a more systematic arrangement and a fuller explanation of the relationships of the groups of substances to one another would probably tend to give the student a simpler, and at the same time a wider grasp of the subject.

CORRESPONDENCE.

THE VOLUMETRIC ESTIMATION OF MANGANESE.

To the Editor of the Chemical News.

SIR,—Messrs. Ibbotson and Brearley, in their letter to the CHEMICAL NEWS, vol. lxxxiv., p. 302, have overlooked the fact that results are given in the original paper on the above subject (*Trans. Chem. Soc.*, 1895, p. 268,) which establish the accuracy of our methods under the given conditions. The point they have now brought forward relating to ferromanganese is therefore already answered.

To sum up the main issues, Messrs. Ibbotson and Brearley have instanced no error in our methods when worked according to the simple instructions contained in the papers. The rare metals occasionally present in small quantities in iron ores, and which pass into the crude iron, cause no appreciable error in the estimation of the manganese. Whilst failing to show that their objection to hydrogen peroxide has any practical bearing except in special steels, they have not met the objection I raised to the employment of ferrous sulphate. The results they quote for their modification are not of the high degree of accuracy established for our method (CHEMICAL NEWS, vol. lxxxiv., p. 210), and until they produce evidence that their modification is at least as accurate as ours, and that it is accurate in the analysis of the special steel, they can hardly expect it to meet with the general acceptance of chemists. They have overlooked the fact that one of our methods, although very simple, clean, and rapid, provides for the analysis of wrought-iron, steel, and pig-iron; they have dealt chiefly with the analysis of special steels. Hydrogen peroxide indicates the presence of the important rare metals in the special steels and no error is likely to occur in the estimation of manganese in these, for special methods of analysis may be applied to them.—I am, &c.

HUGH RAMAGE.

St. John's College, Cambridge,
January 6, 1902.

ON THE MUTUAL ACTION OF ALUMINA AND FERRIC OXIDE AT INCIPIENT WHITE HEAT.

To the Editor of the Chemical News.

SIR,—In connection with Dr. Warth's article on this subject (CHEMICAL NEWS, vol. lxxxiv., p. 305), I should like to draw attention to the effect of ferric oxide upon zinc oxide at a red heat. If the two oxides be ground together in a mortar and heated in a porcelain crucible over a Bunsen burner, the colour of the mixture becomes paler, and a portion of the zinc oxide is rendered insoluble in carbonate of ammonia, a fact which proves, I think, that combination of some sort has taken place between the oxides. There is not the slightest evidence of anything approaching fusion.

The amount of zinc oxide rendered insoluble varies with the temperature employed. A mixture of equal quantities of the two oxides heated for half-an-hour over a Bunsen flame and then extracted with ammonium carbonate, retained 27·5 per cent of the zinc oxide in insoluble form. After further heating for one hour over the blowpipe 42·5 per cent became insoluble. In the case of a mixture of 1 part ferric oxide and 4 parts zinc oxide, the strong red colour of the mixture changed after half-an-hour over the Bunsen flame to a golden-brown.

To a less extent this compound resists the action of sulphuric acid. Where highly pyritic or ferruginous zinc blendes have been roasted "dead," a considerable percentage of the zinc will be found to be insoluble in 10 per cent sulphuric acid, and this percentage will vary with the temperature employed. Unless combination has occurred between the two oxides this insolubility of the zinc oxide is difficult to explain.—I am, &c.,

GILBERT RIGG.

76, Bryn Road, Swansea,
December 31, 1901.

MEETINGS FOR THE WEEK.

MONDAY, 13th.—Society of Arts, 8. (Cantor Lectures). "The Purification and Sterilisation of Water," by Samuel Rideal, D.Sc., F.I.C.

TUESDAY, 14th.—Royal Institution, 3. "The Cell—its means of Offence and Defence—Immunity," by Allen Macfadyen, M.D., B.Sc.

WEDNESDAY, 15th.—Society of Arts, 8. "Elliptographs," by Frank J. Gray, Assoc.M.Inst.C.E., F.R.Met.S.

THURSDAY, 16th.—Chemical, 8. "Myricetin" (Part II.), by A. G. Perkin. "The Colouring-matters of Green Ebony," by A. G. Perkin and S. H. C. Briggs. "An Investigation of the Radio-active Emanation produced by Thorium Compounds" (I.), by E. Rutherford and F. Soddy.

—— Royal Institution, 3. "Recent Excavations at Delphi and in the Greek Islands," by A. S. Murray, LL.D., F.S.A.

FRIDAY, 17th.—Royal Institution, 9. "Interference of Sound," by The Rt. Hon. Lord Rayleigh, M.A., F.R.S., &c.

SATURDAY, 18th.—Royal Institution, 3. "Landmarks in the History of Opera" (with Musical Illustrations), by W. H. Hadow, M.A., Mus Bac.

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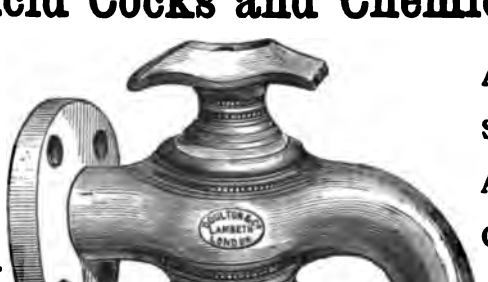

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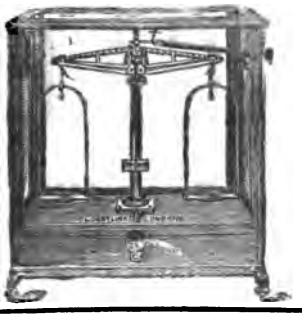

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NOTES ON QUANTITATIVE SPECTRA OF BERYLLIUM.*

By W. N. HARTLEY, D Sc., F.R.S.,
Royal College of Science, Dublin.

In a quantitative examination made in 1885 of all the known methods of separating beryllium from aluminium and from iron, the various precipitates obtained were dissolved and diluted to a known volume corresponding with the amount of bases in solution.

The solutions were spectrographically examined, and the photographs compared with others taken from solutions containing accurately weighed quantities of pure beryllia. The coil used was capable of giving a 5-inch spark in air. In place of a Leyden jar a pane of glass coated on each side with a square foot of tin-foil was used. The electrodes (*Phil. Trans.*, 1884, Part I., p. 49) were Ceylon graphite as in other experiments, the sole impurity in which was a trace of magnesium.

The accompanying tabulated statement gives the wave-lengths of the lines, together with a description of the spectra photographed from solutions of different strengths.

The actual length of the line 2478·1, as rendered by solutions of 0·00001 per cent and 0·000001 per cent strength, is, in the former, 0·07, and, in the latter, 0·05 of an inch. The normal length of the line at this part of the spectrum is 0·22 of an inch. The quantity of substance yielding this spectrum is equivalent to one-millionth of a milligramme of beryllium. As I have pointed out in the case of magnesium (*Phil. Trans.*, 1884, Part II., p. 325), so also is it with beryllium, that the sensitiveness of the spectrum reaction may be increased ten thousandfold by using a larger coil and more powerful condenser, but leaving the striking distance between the electrodes unaltered. The coefficient of complete extinction was, therefore, practically not attainable for all the lines, or, in other words, the sensitiveness of the reaction is almost without limit.

It will also be seen from my description of the spectra, which have been quite recently re-examined, that the coefficient of extinction of the two lines λ 3130·3 and 2478·1 had not been reached by the dilution specified.

A number of thin sections of the Dublin granite containing microscopic crystals of hexagonal form were examined some years ago. The crystals were supposed to be apatite, but a very carefully executed analysis disclosed the fact that the proportion of phosphoric acid contained in 20 grms. of the rock was almost infinitesimal, and that on warming thin sections, in which the hexagonal crystals were visible, with nitric acid and ammonium molybdate, no deposit of yellow ammonium phosphomolybdate could be detected by the microscope. A spectrographic analysis showed that these crystals were really beryls, and similar crystals a millimetre in length were picked out of the granite. They were found to contain between 10 and 11 per cent of beryllia. Since then beryllia has been separated from the alumina of felspar obtained from the granite in Glen Cullen in proximity to a vein of coarse granite in which beryls were found by Dr. John Joly.

From numerous experiments on the analytical processes employed in the separation of beryllia from alumina it was found that it remained combined with the sesquioxide bases in so persistent a manner as to lead to the belief

* A Paper read before the Royal Society, December 5, 1901.

Strength of Solutions or Degrees of Dilution shown by Per cent of Beryllium.

Beryllium wave-lengths (Rowland's scale).	1 per cent.	0·1 per cent.	0·01 per cent.	0·001 per cent.	0·0001 per cent.	0·00001 per cent.	0·000001 per cent.
3322·3	A continuous line.	Half of the line is much weakened.	A very short line.	A faint indication.	Extinct.	Extinct.	Extinct.
3130·3	A continuous line.	A continuous line.	One-half of the line weakened.	One-half of the line weakened.	Line shorter by one-half.	One-half the line still strong.	Nearly one-half, still strong.
2649·8	A continuous line.	Half of the line is weakened.	A very short line.	Very short line.	A dot merely.	A dot.	A dot.
2493·6	A continuous line.	Half of the line is weakened.	Very short line.	Very short line.	A very faint dot.	A dot, scarcely visible.	A dot, scarcely visible.
2478·1	A continuous line.	A continuous line.	A short line.	A very fine short line.	Very short line.	A very fine short line.	A very fine short line.

that ordinary alumina might be found more often than not to contain traces of beryllia, particularly as there is no easily applied chemical test for detecting its presence in small quantities, nor a simple means of separating it. It has, however, been found that such is not the case, though gallium has been ascertained to be present in almost all minerals which contain aluminium. As they belong to the same group, the two elements aluminium and gallium may be expected to form isomorphous mixtures, which would account for their being so constantly associated in nature; but the position of beryllium in the periodic system of classification shows that a similar behaviour with that element is scarcely probable.

ON AN INTEGRAL METHOD FOR THE DESTRUCTION OF ORGANIC MATTERS, APPLICABLE TO THE SEARCH FOR MINERAL POISONS, ESPECIALLY ARSENIC AND ANTIMONY.

By G. DENIGÈS.

Besides the method of destruction by means of a large proportion (30 per cent) of bisulphate of potassium, that G. Pouchet has applied to the detection of small quantities of lead buried in a large mass of organic matter, attempts have been made of late years to integrally destroy such matters, so as to be able more especially to introduce the whole residue from the operation directly into a Marsh's apparatus for the search for arsenic and antimony.

In this way Nikitin and Igefsky dried the organs at 110—115°, and then heated the material with about ten times its weight of concentrated sulphuric acid in a Kjeldahl flask. It requires at least two or three days, and sometimes even four or five days of continuous boiling with acid to obtain complete decolouration.

It is hardly necessary to point out the extreme impracticability of this method; besides the very considerable time required for its performance, the great and dangerous risk of breakage which is always present, and the enormous quantity of sulphuric acid which has to be used (2 kilogrms. for only 200 grms. of organic matter), we are working with a reagent which for a long while remains strongly reducing, and thus we run the risk of losing arsenic in the form of As_2O_3, especially in the presence of chlorides. Finally, besides these objections, if the process could even be properly used for an isolated case, it could not be made applicable to a systematic series of destructions.

We have succeeded, without the risk of any loss, in destroying various anatomical samples in a few hours by making use of the following process which is absolutely general in its application, and can even be used for the destruction of the cacodylic molecule which has such great resisting power to other methods of attack; this method, therefore, appears to us capable of being of great service in toxicology and in the elimination of mineral poisons.

This method utilises in the first place the curious oxidising action of the salts of manganese in a nitric medium, discovered by Villiers and applied by that chemist, but under conditions slightly different to ours, to the partial destruction of organic matters, and terminates by the combined action of sulphuric and nitric acids. The following is a detailed description of the process:—200 grms. of the material, in fairly large pieces, are introduced into a porcelain dish of 2 litres capacity, with 200 c.c. of nitric acid at about 40° B. (density 1·39 to 1·40), and 5 c.c. of permanganate of potassium at 2 per cent; this is heated over a Bunsen burner, the dish resting on a sheet of iron 2 or 3 m.m. thick, 11 to 12 c.m. diameter, and perforated at the centre with a hole 4 c.m. diameter.

After from a quarter to half-an-hour, according to the state of division of the mass and the nature of the organs (it is more rapid with muscle, but less so with the viscera, liver, &c.), the disaggregation is complete, and the froth at first formed gives way to tranquil boiling. If the froth, which is particularly abundant in the case of parenchymatous (liver, kidneys) organs, on account of the urea or ammoniacal products which they may contain, and especially with skins and hair which are rich in carbonate of lime, if this froth is in any danger of running over the sides of the dish, it must be broken up with a stirring-rod, or the flame should be withdrawn for a time; this is often necessary with epithelious organs, more particularly skins and hair. In the latter case, the length of time required for the disaggregation will slightly exceed the limits we have mentioned above.

On reaching this stage the mass is placed in a 1 litre porcelain dish, the large dish is rinsed with 100 c.c. of nitric acid at 40° B., which is heated to about 50—60° in the dish itself, and then poured into the 1 litre dish; the same is repeated with 100 c.c. of warm water for the final rinsing of the large dish.

All the liquid, as well as the supernatant fat, being collected together in the 1 litre dish, this latter is covered with a large glass funnel, the edge of which reaches to the commencement of the lip of the dish, and of which the stem has been cut down to about 1 or 2 c.m., leaving an opening of about 15 to 20 m.m. diameter; the whole is then raised to gentle boiling, when a mixture of nitrous fumes, nitrogen, and carbonic acid is given off.

The heat is continued for at least two hours, as it is necessary to reduce the volume to a little less than 100 c.c.; if there is time it is preferable to lower the flame, and to allow the attack to continue for four or five hours. In any case care must be taken never to evaporate so far that the mixture commences to blacken; by stopping when the volume of the residue is still about 70 or 80 c.c. this can be prevented.

If, however, this should happen during the course of the operation, it would be necessary to stop at the first symptom, of browning, and to add to the mass 10 to 20 c.c. of nitric acid, and then heat again until the liquid is entirely clarified.

When the desired volume is reached (70 to 80 c.c.) the funnel and the flame are both removed, then, without any time for cooling, 100 c.c. of pure sulphuric acid are added rapidly, and stirred with a glass rod;* reddish-brown fumes are rapidly evolved, then, as they cease coming off, the mass becomes brown. We then wait about two minutes from the time when this colouration appears, and add 5 c.c. of nitric acid in a fine stream, which must be run in from a pipette at the centre and close down to the surface; this operation is repeated four times in all, using about 20 to 25 c.c. of nitric acid. After the last addition the whole is heated strongly for four or six minutes, so that the sulphuric acid will thoroughly attack the supernatant fatty matters; the flame is then removed, and then, three times in succession, at two minutes interval, 5 c.c. of nitric acid are added, operating as described above. This done the funnel is replaced, and heat again applied, but always with the sheet-iron disc under the dish so as to ensure the sulphuric acid boiling.

From this moment, every two or three minutes, we pour, a drop at a time, 50 to 60 drops of nitric acid at 40° B., using a capillary funnel passing through the hole in the top of the inverted large funnel, and letting the end reach to about 1 c.m. of the surface of the material operated upon. After each addition the funnel should be raised a little to prevent superheating and cracking from the cold nitric acid when the dropping is not quite continuous.†

* When the fatty residue is very considerable, we are sometimes, but only in *exceptional* cases, obliged to exceed this quantity of acid; the important point being to leave the mass fluid, even after attacking with sulphuric acid and after intense blackening.

† Time can be saved and the operation made less tedious by adding the nitric acid in a continuous manner with the aid of a

After about ten to fifteen additions of acid, or even more in the case of very fat viscera, the residual liquor, even when strongly heated after all the nitric acid has been driven off, takes first a reddish-yellow and then a bright yellow colour. The excess of sulphuric acid is then driven off by evaporation, so as to bring the final volume of the residue down to 10 or 15 c.c. During the evaporation 50 or 60 drops of nitric acid must again be added, at least four or five times, through the capillary funnel. After cooling, the residue which ought to be colourless or at most barely yellow, is treated with 100 c.c. of water; nitrous fumes are generally given off owing to the hydrolytic decomposition of a nitro-sulphuric acid formed during the course of the operation, and of which the presence is a certain, if not a necessary indication, of the complete destruction of the organic matter. After boiling to completely drive off these fumes, the whole is again cooled, and enough distilled water added to give a dilution of a tenth, by volume, of the final acid residue, which should have been carefully measured before the addition of water.

In this manner a perfectly colourless liquid is obtained which contains integrally the whole of the arsenic, antimony, and mercury present in the original substance under examination, as we have proved by our experiments, on numerous occasions.

Very frequently (even apart from bone and tissues rich in calcareous material, such as epidermic products) the liquid holds in suspension a small quantity of crystalline mineral residue, generally consisting of sulphate of lime, and sometimes of ferric sulphate with anatomical fragments rich in ferruginous matter (liver, &c.); in the latter case the boiling with sulphuric acid is sometimes attended with bumping, the crystalline grains being much less soluble than the sulphate of lime in concentrated acid.*

These mineral residues, unless they are very abundant, when they can be separated by filtration through a plug of cotton-wool or gun-cotton, in no way interfere with the direct introduction of the liquid containing them into a Marsh's apparatus; neither do the extremely faint traces of nitric products which may be present in the liquid; they can, however, be removed if desirable before the final addition of water by the aid of oxalic acid, urea, or sulphate of ammonium added directly in the form of powder, to the colourless, boiling sulphuric residue.

The above method is absolutely general in its application; we shall now, in addition, proceed to point out the slight variations which can be introduced for the destruction of such bodies as glycerin, cacodylic products, &c., which are reputed difficult to destroy, or in other important instances such as bone, hair, skin, milk, urine, &c.

Destruction of Epidermoid Materials and Bone.

With hair treated by the general method, after a few minutes in the cold, a lively effervescence takes place; the attack goes on by itself without the aid of heat, and the material is practically dissolved after fifteen or twenty minutes. It is then heated; the amount of nitric acid used should be, for the same weight of material, double the ordinary proportion. Liquefaction once obtained the operation is completed as above.

With skins, analogous phenomena are observed, and the manipulation is the same.

With bone, when once the nitric attack is finished and the reduction of the volume to 70 or 80 c.c. effected, the whole is cooled, filtered through cotton-wool or gun-cotton, washed with dilute nitric acid, retaining the fat, which must be washed with great care. The filtrates are diluted with a large quantity of water, precipitated with a slight excess of sulphuric acid, and filtered through cotton-wool or gun-cotton, or even glass-wool. The precipitate of sulphate of lime is washed until it is perfectly white and the washings colourless; the filtrates are then again concentrated down to 70 or 80 c.c., the filtered fat added, and 100 c.c. of sulphuric acid (for 200 grms. of bone); it is then heated, and the operation completed as in the general case, always taking care not to push the evaporation too far, and to leave, besides the phosphoric acid, 10 to 15 c.c. of *sulphuric acid* in the residue, which can be diluted proportionally.

Destruction of Milk.

To 400 c.c. of this liquid, placed in a 1 litre porcelain dish, we add 200 c.c. of nitric acid at 40° B., 5 c.c. of 2 per cent permanganate of potash, raise to gentle boiling, and evaporate down to 70 or 80 c.c., after having covered the dish over with the large funnel with a short stem already described; while still warm 100 c.c. of sulphuric acid are added, and the operation completed as in the general case. In this manner we have easily succeeded in detecting 0·5 m.grm. of mercury in 1 litre of milk by using Merget's method, which consisted of immersing for twenty-four hours in the liquid diluted with water, a copper-wire which has been washed in water, alcohol, and ether, and then dried, and then placing it in contact with paper soaked in ammoniacal nitrate of silver for half-an-hour or an hour.

Destruction of Urine.

500 c.c. of urine are boiled in a 1 litre porcelain dish with 100 c.c. of nitric acid at 40° B., and 5 c.c. of 2 per cent permanganate of potash. The whole is covered over with a large funnel as soon as the froth which is first formed disappears.

When the volume is reduced to 100 or 150 c.c. we add a further 100 c.c. of nitric acid and heat again until the volume is reduced to 60 or 80 c.c.; then, while still warm, 25 c.c. of pure sulphuric acid are added (50 c.c. in the case of diabetic urines); this is then heated until strong blackening takes place and white fumes are given off; the destruction and decolouration of the mass are then completed by nitric oxidation as in the general case.

We have observed that the presence of a large proportion of chlorides in urine (up to 12 grms. per litre, that is to say 6 grms. in the ordinary sample of 500 c.c.) does not lead to any loss of arsenic under these conditions. Still, if we wish to do away with the influence of these salts, we must work on a sample never containing more than 5 or 6 grms. of chlorides, and dilute to 500 c.c.; or still better—to 700 c.c. of urine we add 20 c.c. of nitric acid and as many times 1·5 grms. of powdered nitrate of silver as there are grms. of chlorides (expressed as NaCl) per litre of the urine examined. This is well shaken several times, allowed to settle, and 5·15 c.c. of the clear liquid decanted or filtered off, representing 500 c.c. of the urine under examination, which is then treated as above

Destruction of Wine.

In a two-litre dish, one litre of red or white wine is reduced by boiling to about 300 c.c., then, removing the flame, we add 5 c.c. of 2 per cent permanganate of potash in a very fine stream while continually stirring, and 100 c.c. of nitric acid at 40° B. When the effervescence which is generally produced has abated, the mixture is placed in a one-litre dish and covered with the large funnel with a short stem. It is then boiled continuously and steadily until the volume is reduced to 50 or 60 c.c.; without removing the flame 30 c.c. of pure sulphuric acid are then added. After the blackening of the mass and expulsion of the nitrous fumes, nitric acid is added in several portions and the operation is continued and terminated as in the general case.

When we have to deal with a sweet wine, the proportions of nitric and sulphuric acids is doubled.

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syphon fitted with a glass tap, having the short end plunged into the acid and the long end drawn out fine, passing 1 drop every two seconds through the capillary end; this can easily be adjusted by means of the tap.

* It is obvious that when the substances destroyed contain a sufficient quantity of lead, barium, or strontium, these metals will be found, at least in part, in the residue in the state of insoluble sulphates.

Destruction of Glycerin.

One hundred grms. of glycerin are placed in a 1 litre porcelain dish, with 500 c.c. of a mixture of equal volumes of nitric acid at 40° B., and water; then add 5 c.c. of a 2 per cent solution of permanganate of potash. Heat over a Bunsen burner, using the perforated iron plate.

Between 90° and 95° a lively reaction takes place with an abundant disengagment of reddish-brown nitrous fumes. The flame is immediately removed, but in spite of that, the energetic chemical reaction quickly raises the temperature to above 100°. When it has cooled down to about 90°, heat is applied again by means of a very small flame, so as to keep the temperature between 90° and 95°, without, however, surpassing the latter point; it is then covered with the large funnel described above.

The reddish fumes diminish considerably, and at last a moment arrives when the attack goes on regularly with tranquil effervescence and barely noticeable disengagement of nitrous products.

When the volume of the liquid is reduced to 75 or 100 c.c., 500 c.c. of the already mentioned hydronitric mixture are added. Heat is again applied until abundant reddish fumes come off, the funnel is replaced, and the temperature brought to and maintained at about 95° until the volume is again reduced to about 100 c.c.

At this point 25 c.c. of sulphuric acid are added, while still heating, and the funnel put on again. If nitrous fumes come off very freely the flame is removed immediately to allow the action to slacken. If the fumes do not come off very abundantly the heating is continued.

In both cases, however, a moment arrives while still heating, when the evolution of nitrous fumes ceases and the mass becomes decolourised; soon after it becomes yellow, then brown, and gives off an odour of caramel.

When it has turned black the decolouration must be effected by means of nitric acid and the operation completed as in the general case. The destruction is effected in a most perfect manner.

Destruction of Methylene Blue.

Twenty-five grms. of methylene blue are heated in a 1 litre porcelain dish with 75 c.c. of nitric acid at 40° B., 25 c.c. of water, and 5 c.c. of 2 per cent permanganate of potash, the whole being covered with the large funnel.

After boiling for half-an-hour, 50 c.c. of nitric acid are added, being poured round the sides of the dish.

Heat is applied until the blue colouration gives way to a reddish tint and the volume is reduced to about 50 c.c.

At this moment 25 c.c. of concentrated sulphuric acid are added, and the operation terminated as in the general case.

Destruction of Cacodylic Molecule.

If we suspect the presence of cacodylic products in the organic substances it is sought to destroy, after having operated as described in the general case, or in any one of the particular ones just described, and having arrived at a final volume of about 15 c.c. in the latter part of the operation, we add 5 or 6 grms. of pure nitrate of potash and heat until the excess of free sulphuric acid is driven off, and fused bisulphate of potash formed. After cooling, the saline mass is taken up with 100 c.c. of boiling 10 per cent sulphuric acid in water; this is again allowed to cool after complete solution.

By thus destroying 200 grms. of horseflesh containing 0·08 grm. of pure anhydrous cacodylate of soda the whole of the arsenic was recovered, either by the ring method, or by the nitrate of silver method, without any garlic odour being noticed during the destruction and subsequent hydruration. Further, the quantity of bisulphate of potash present in the substance is too small to interfere with the direct introduction of the material into the Marsh's apparatus and form crystals of potasso-zincic sulphate on the zinc.

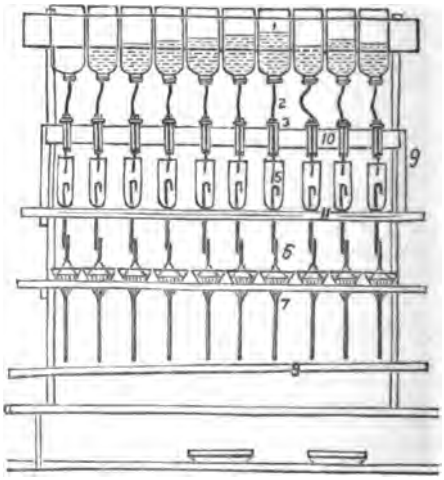
We may add, in conclusion, that if it is desired to employ the process now described and to be certain of success, it suffices to make one preliminary trial with either beef or horseflesh, and to remember that it is more especially the additions of nitric acid to the sulphuric acid heated sufficiently to emit thick white fumes, which effect the complete destruction of the material. The use of the funnel, as described, is also indispensable; it can easily be handled with a strong pair of spring tongs with which the stem is held, and which for this reason therefore must not be cut too short.

It is well understood that the operations should always be carried out in a glazed fume cupboard with a strong draught, with just a small opening giving access to the apparatus.—*Bull. Soc. Chim.*, Series 3., xxv., No. 22.

AN AUTOMATIC FILTER-WASHER.

By J. M. PICKEL.

The apparatus shown in the accompanying cut consists of a battery of ten washers. The parts of each washer are:—A reservoir, 1, to contain the liquid with which the washing is done. A rubber tube, 2, provided with two thumb-screw clamps, 3, leading from the reservoir to the delivery vessel. A delivery vessel, 5, provided interiorly with a small syphon, which delivers the washing liquid intermittently in small portions on the substance being washed. A larger funnel, 7, containing the filter. A smaller inverted funnel, 6, covering the substance. The object of this funnel is, while it prevents spattering, to throw the liquid around over the edge of the filter-paper, thus insuring that it be washed from the top downward at each delivery. It should be of such a diameter as almost, but not quite, to cover the filter-paper. A trough, 8, which conveys the washings, in case they are not wanted, into the waste pipe. A supporting frame, 9, 10, 11.



The apparatus is operated thus:—The substance to be washed having been placed on the filters and covered with the inverted funnels, and the lower clamps having been closed, each reservoir is provided with a measured quantity of liquid adequate to the washing. To facilitate the measuring, the reservoirs should be graduated once for all. By means of the lower clamp discharge the liquid rapidly into the delivery vessel until the syphon overflows, then regulate (by the *lower* clamp) the flow, so that the liquid shall fall, drop by drop, into the delivery vessel at such a rate that the syphon shall not overflow till after all the liquid has passed out of the filter. This regulation insures the intermittent washing of the filter with entirely fresh portions of liquid at each delivery, and, with a little

practice and patience, can be accomplished in about ten to fifteen minutes for all ten of the washers. After the apparatus is once regulated, it requires no further attention—may be left going over night.

The upper clamp is not necessary, but is at times convenient; for example, if for any reason it is desired to stop the flow of liquid before the washing is completed, this is effected preferably by the upper clamp, thus leaving the "*set*" of the lower clamp undisturbed. The efficiency of the machine depends upon the ease and accuracy with which it may be regulated. The screw of the regulating clamp must, therefore, have a fine thread; the clamp must be firmly fixed in place, otherwise the merest touch will sometimes change the rate of flow. The washer at the extreme left of the cut shows a method of securing this fixedness. The clamps and attachment are there turned through an angle of 90°. A piece of wood about two inches long and of rectangular section passes through the clamp. Transversely across the back of the piece of wood are shallow grooves in which the clamps fit. These pieces are firmly attached by screws to the frame-piece marked 10. Small wire brads, one just above and one just below the clamp, and each slightly pressing against it, are driven a short way into the wood, completing thus the steadiness of the clamps. The arrangement here described is satisfactory; but doubtless a tube and glass cock, capable of delivering small drops (after the manner of a good burette) would be preferable.

The quantity of water delivered at each discharge of the syphon should, of course, be sufficient to cover the substance, but not enough to flood the filter to overflowing. The capacity of the delivery vessel may be varied by adding to or taking from it, small glass beads, coarse sand, or fragments of glass. It is evident that, if the syphon is on the outside of the delivery vessel (after the manner of a Soxhlet extractor), the capacity of the vessel may easily be varied from its greatest volume down to an almost vanishing quantity by shoving down into it a tube closed at its lower end (a test-tube for example); a small plug of rubber wedged in between would hold the tube at any desired level. A Soxhlet can be utilised as a delivery vessel; one in possession of the writer whose normal maximum delivery is 43 c.c. can, in the manner just described, be instantly made to deliver any lesser quantity down to 7 c.c.

As is seen from the cut, the delivery vessels in the apparatus here described are what are known as carbon filters, for Gooch filters. Their dimensions are, for the large end, about 2 to 3 inches in length and 1¼-inch internal diameter, small or stem end, about 2 inches in length and ¼-inch internal diameter. The long arm of the syphon passes through the stem, and is made water-tight by a short piece of rubber tubing. They were regulated once for all to deliver about 10 to 15 c.c. The syphons should be well made, particular care being taken that they are not flattened, or contracted, at the bend A syphon thus contracted requires, especially when it becomes somewhat soiled internally, and does not readily wet, considerable pressure to force the water over; it will therefore vary considerably in the volume of liquid that it delivers at an overflow. For this reason also a narrow tall delivery vessel is to be preferred to one of larger diameter—there is less variation in the quantity of liquid delivered at each overflow of syphon in the former than in the latter. A diameter (internal) of not over an inch—of even three-quarters of an inch or less if practicable—is to be recommended. I especially call attention to this, because I found that some of my delivery vessels, which were set to deliver about 10 c.c., would, owing to the causes here detailed, sometimes cause my filters to overflow (I was using quite small, 9 c.m. filters). The reservoirs in this apparatus were made by cutting off, near the bottom, pint bottles. Small percolators would be neater.

The combination here described as an automatic filter-washer is, so far as the writer knows, new. At any rate, it was new to him, and devised by him for washing water-soluble nitrogen (more especially nitric and ammoniacal nitrogen) out of mixed commercial fertilisers. It was used the past winter and spring for that purpose in the analysis of several hundred samples, and was found entirely satisfactory and a valuable labour-saver. Two nitrogen determinations were made in each case, one of total nitrogen, the other of residual nitrogen after washing with 300 c.c. of distilled water.

There would seem to be no reason why this washer might not be used, with equal advantage, to take water-soluble phosphoric acid out of commercial fertilisers. A comparison of its work in this matter with the method of washing commonly practised, was made with the following result on ten different fertilisers :—

Per cent.	Water soluble P_2O_5.		Difference.
1	6·65	6·42	0·23
2	4·55	3·78	0·77
3	9·51	8·92	0·59
4	9·31	8·75	0·56
5	8·04	7·88	0·16
6	9·24	8·65	0·59
7	5·97	6·10	—
8	6·10	5·68	0·42
9	5·06	4·72	0·34
10	6 03	5·42	0·58

The percentages in the first column were obtained in the washings from the automatic washer; those in the second column were obtained in the usual way by directing a jet of water on the substance, and thoroughly stirring up at each washing. Two grms. of fertiliser were taken in each case, and the same quantity (or about the same quantity), a little less than 300 c.c. of water, was used for washing in each case. In all except one case, the automatic washer took out more phosphoric acid than the usual method. This is all the more remarkable inasmuch as the washer does not *stir* the substance to any appreciable extent. The explanation of the difference is perhaps to be found in this circumstance; the washer delivered the water in small portions of about 10 c.c. at a time, whereas in the other case 30 to 40 c.c. were used, the filter being larger; it thus came about that in the first case the samples got three to four times as many separate washings as in the second. There was this further circumstance:—The machine, once set going, keeps up the work to the end, thus finishing the operation in a shorter time than did the hand washing, where the chemist was diverted by other operations which he was looking after at the same time. Such being the case, the phosphates were in contact with the water a shorter time in the first than the second instance, and the monocalcium phosphate had less opportunity (less time) to "*revert*."—*Journal of the American Chemical Society*, xxiii., No. 8.

Methods of the Formation of Peroxide of Lead.—G. Kasner.—The author has already shown that metaplumbate of lime gives, through the action of heat, peroxidised compounds of lead, of which the lime salts are veritable perplumbates. The orthoplumbate containing water of crystallisation, $Ca_2PbO_4.4H_2O$, gives the same result. The formation of these peroxidised compounds should not be attributed to a fixation of free oxygen, but to an internal oxidation; that is to say, to a fixation on the lead of oxygen furnished by the molecule of the heated salt. The peroxide of lead is, in fact, produced in the complete absence of free oxygen; for example, in the presence of inert gases or even in vacuo. In the preparation of this peroxide, it is necessary to prevent a too prolonged action of heat, for fear of destroying the molecule formed, with the liberation of the inactive oxygen.—*Arch. d. Pharm.*, vol. ccxxxviii., p. 449.

ON THE RELATION OF THE VISCOSITY OF MIXTURES OF SOLUTIONS OF CERTAIN SALTS TO THEIR STATE OF IONISATION.*

By JAMES BARNES, B.A., Dalhousie College, Halifax, N.S.

(Continued from p. 5).

Determination of the Ionisation Coefficients at 25° C. for Simple Solutions.

The ionisation coefficient for a simple solution is taken to be the ratio of the specific molecular conductivity to the specific molecular conductivity at infinite dilution. Before this ratio could be found for 25° C. it was necessary to determine the values of the specific conductivity at 25° from Kohlrausch's values at 18° (Kohlrausch und Holborn, "Leitvermögen der Elektrolyte," 1898, p. 159, Table 2) by means of Déguisne's (*loc. cit.*) and Kohlrausch and Grotrian's temperature coefficients (Kohlrausch und Holborn, *loc. cit.*, p. 145, Table 1). The concentrations of solutions of the salts for which Kohlrausch gives conductivity values did not in all cases correspond to the concentrations of solutions for which Reyher and Wagner determined the viscosity. In such cases (concentrations 0·25 and 0·125), the values of the specific conductivities at 25° were obtained by interpolation.

Table III. gives both the values of the specific conductivity at 25° C. determined as above from the values at 18°, and the calculated ionisation coefficients at 25°. Only those coefficients are given which are necessary in the calculation of the viscosities. Under copper sulphate are given a few conductivity values of higher concentration, these being necessary for the determination of the ionisation coefficients in the mixtures by the method used. The concentrations are expressed as in Table I., and conductivities in terms of 10^{-4} times Kohlrausch's new unit.

Determination of the Ionisation Constants.

Table IV. gives the values of the ionisation constants (k and l) determined by the method of least squares from the data given in Tables III. and V., the observed values of the viscosity of the four solutions of each salt being used. The relative magnitude and the sign of the ionisation constants would seem to show that the undissociated molecules exert the greater influence in increasing the viscosity, while the free ions have in some cases a diminishing effect.

Results of Calculations on Simple Solutions.

Table V. gives a comparison of the calculated and observed values of the viscosity of simple solutions, the calculated values being determined by expression (1) with the ionisation coefficients and ionisation constants, as given in the above tables. In this table, all the viscosity results are relative to water at 25° C., and the concentrations are expressed as in Table I.

As both Reyher and Wagner regard their results as affected by a possible error of about ± 3 in the third decimal place, it is seen that the agreement between the calculated and observed values for all the salts except copper sulphate is very satisfactory, the differences being well within the limit of experimental error. In the case of copper sulphate, the agreement is not so satisfactory. But it was noticed on plotting the observed values against the concentration that the points do not lie on a smooth curve, and that the point corresponding to the concentration 0·5 is at quite a distance from this curve, which leads one to think that this observed value cannot be correct. The poor agreement in this case might also be partly due to the doubtful value of the specific molecular conductivity at infinite dilution used. Thus it seems that for all the salts examined, copper sulphate perhaps excepted, expression (1) gives the viscosity of a solution within the limit of experimental error throughout a concentration range of 1·0 to 0·125.

Table III.

Concentration.	Specific conductivity at 18° C.	Specific conductivity at 25° C.	Ionisation coefficients at 25° C.
	NaCl.		
1·0	744·0	862	0·672
0·5	404·5	469	0·732
0·3	255·6	296	—
0·25	—	252	0·786
0·2	176·4	205	—
0·125	—	131	0·817
0·1	92·5	107	—
	KCl.		
1·0	982·0	1128	0·743
0·5	511·5	588	0·774
0·3	315·9	363	—
0·25	—	308	0·811
0·2	215·4	248	—
0·125	—	159	0·838
0·1	111·9	129	—
	$\frac{1}{2}BaCl_2$.		
1·0	703	811	0·566
0·5	388	448	0·624
0·3	249	287	—
0·25	—	245	0·684
0·2	173·4	200	—
0·125	—	130	0·726
0·1	92·2	106	—
	$\frac{1}{2}K_2SO_4$.		
1·0	718·0	827	0·528
0·5	393·5	453	0·578
0·3	253·2	292	—
0·25	—	251	0·640
0·2	177·8	205	—
0·125	—	135	0·690
0·1	95·9	111	—
	$\frac{1}{2}Na_2SO_4$.		
1·0	508·0	591	0·443
0·5	298·5	347	0·520
0·3	199·8	230	—
0·25	—	201	0·604
0·2	142·8	166	—
0·125	—	110	0·662
0·1	78·4	91·4	—
	$\frac{1}{2}CuSO_4$.		
2·631	458	534	—
2·194	421	489	—
1·0	258	297	0·209
0·5	154	177	0·249
0·3	106·5	122	—
0·25	—	107	0·302
0·2	78·4	89·9	—
0·125	—	61·7	0·347
0·1	45·0	51·6	—

Table IV.

Electrolyte.	k.	l.
NaCl	+0·11213	+0·089765
KCl	+0·30645	−0·12289
$\frac{1}{2}BaCl_2$	+0·20327	+0·061009
$\frac{1}{2}K_2SO_4$	+0·21347	+0·0088236
$\frac{1}{2}Na_2SO_4$	+0·30418	+0·13348
$\frac{1}{2}CuSO_4$	+0·46500	−0·058144

* *Transactions of the Nova Scotian Institute of Science*, x., p. 113.

TABLE V.—*Viscosity at 25° C.*

Concentration.	Observed value.	Calculated value.	Difference.
	NaCl (Reyher).		
1·0	1·0973	1·0971	−0·0002
0·5	1·0471	1·0479	+0·0008
0·25	1·0239	1·0236	−0·0003
0·125	1·0126	1·0117	−0·0009
	KCl (Wagner).		
1·0	0·9872	0·9874	+0·0002
0·5	0·9874	0·9871	−0·0003
0·25	0·9903	0·9896	−0·0007
0·125	0·9928	0·9933	+0·0005
	$\frac{1}{2}BaCl_2$ (Wagner).		
1·0	1·1228	1·1228	±0·0000
0·5	1·0572	1·0572	±0·0000
0·25	1·0263	1·0265	+0·0002
0·125	1·0128	0·0125	−0·0003
	$\frac{1}{2}K_2SO_4$ (Wagner).		
1·0	1·1051	1·1054	+0·0003
0·5	1·0486	1·0476	−0·0010
0·25	1·0206	1·0206	±0·0000
0·125	1·0078	1·0090	+0·0012
	$\frac{1}{2}Na_2SO_4$ (Wagner).		
1·0	1·2291	1·2286	−0·0005
0·5	1·1058	1·1078	+0·0020
0·25	1·0522	1·0502	−0·0020
0·125	1·0235	1·0239	+0·0004
	$\frac{1}{2}CuSO_4$ (Wagner).		
1·0	1·3580	1·3556	−0·0024
0·5	1·1603	1·1675	+0·0072
0·25	1·0802	1·0767	−0·0035
0·125	1·0384	1·0354	−0·0030

(To be continued).

THE CONSTITUTION OF TOURMALINE.*

By F. W. CLARKE.

(Continued from p. 14).

BETWEEN the magnesium tourmalines and the iron tourmalines the closest analogy exists, and the identity of type is absolute. Taking except when otherwise specified, the analyses by Riggs, all the iron tourmalines reduce to mixtures of the following isomorphous molecules:—

A. $Al_5(SiO_4)_6(BO_2)_2.BO_3(AlOH).Fe_4H_4$,
B. $Al_5(SiO_4)_6(BO_2)_2.BO_3Ca.Fe_4H_4$,
C. $Al_5(SiO_4)_6(BO_2)_2.BO_3NaH.Al_3NaH_2$,
D. $Al_5(SiO_4)_6(BO_2)_2.BO_3NaH.Al_2Na_2H$,
E. $Al_5(SiO_4)_6(BO_2)_2.BO_3NaH.Al_3Na_3$

Molecules C, D, and E are evidently identical, except in the varying replacements of sodium by hydrogen. A and B are similarly alike, so that actually only two fundamental compounds are assumed. From the commoner iron tourmalines lime is practically if not quite absent; and these may be interpreted very nearly as mixtures of A and C, such as A_8C_5, A_7C_5, &c. If we take the minute quantities of lime into account, the black tourmalines from Brazil and from Stoney Point, North Carolina, correspond to $A_{13}B_2C_9$; that from Auburn, Maine, to $A_{35}B_2C_{27}$, and that from Paris, Maine, to $A_{10}B_1C_9$. It will be noticed that the molecule A is in excess of the other two—a condition which fits the analyses, but which is incompatible with the formula proposed by Penfield and Foote. To satisfy the latter, the number of A molecules should be exactly equal to B+C, giving the ratio Si_4O_{21} or $Si_6O_{31.5}$. The analyses in question are as follows:—

	Brazil.	Stoney Point.	Auburn.	Paris.
SiO_2	34·63	35·56	34·99	35·03
B_2O_3	9·63	10·40	9·63	9·02
TiO_2	—	0·55	—	—
Al_2O_3	32·70	33·38	33·96	34·44
Fe_2O_3	0·31	—	—	1·13
FeO	13·69	8·49	14·23	12·10
MnO	0·12	0·04	0·06	0·08
MgO	2·13	5·44	1·01	1·81
CaO	0·33	0·53	0·15	0·24
Li_2O	0·08	trace	—	0·07
Na_2O	2·11	2·16	2·01	2·03
K_2O	0·24	0·24	0·34	0·25
H_2O	3·49	3·63	3·62	3·69
F	0·06	—	—	—
	99·52	100·42	100·00	99·89

The reduced analyses and their comparison with the calculated composition is as follows:—

	Found. Stoney Point.	Found. Brazil.	Calculated. $A_{13}B_2C_9$.	Found. Auburn.	Calculated. $A_{35}B_2C_{27}$.	Found. Paris.	Calculated. $A_{10}B_1C_9$.
SiO_2	34·10	34·26	34·27	34·75	34·48	34·70	34·62
B_2O_3	9·98	9·54	9·99	9·56	10·06	8·94	10·10
Al_2O_3	32·37	32·54	32·36	33·73	33·28	34·82	33·60
FeO	17·33	17·45	17·14	15·99	15·95	15·30	15·23
CaO	0·52	0·33	0·45	0·15	0·17	0·25	0·27
Na_2O	2·22	2·40	2·22	2·23	2·50	2·33	2·67
H_2O	3·48	3·48	3·57	3·59	3·56	2·66	3·51
	100·00	100·00	100·00	100·00	100·00	100·00	100·00

Here, again, the agreements between analysis and theory are as close as could be reasonably expected. The same thing is true of the black tourmalines from Haddam Neck, Connecticut, and Nantic Gulf. Using letters to represent the several molecules, as above, the Haddam mineral is sharply represented by $A_4B_1D_2$, and that from Nantic Gulf by $A_6B_7C_3E_1$. Here is the comparison:—

	Haddam Neck.			Nantic Gulf.		
	Found.	Reduced.	Calc.	Found.	Reduced.	Calc.
SiO_2	34·95	33·78	33·67	35·34	33·34	33·60
B_2O_3	9·92	9·60	9·82	10·45	9·86	9·80
TiO_2	0·57			0·40		
Al_2O_3	31·11	30·74	30·67	30·49	29·01	28·84
Fe_2O_3	0·50			—		
FeO	11·87	19·31	19·24	8·22	20·46	20·55
MnO	0·09			trace		
MgO	4·45			7·76		
CaO	0·81	0·78	0·75	2·32	2·19	2·15
Na_2O	2·22	2·30	2·48	1·76	1·74	1·70
K_2O	0·24			0·15		
H_2O	3·62	3·49	3·37	3·60	3·40	3·36
	100·35	100·00	100·00	100·49	100·00	100·00

To the lithia tourmalines, as analysed by Riggs, a similar set of formulæ apply, although the comparison between fact and theory is not quite so close as in the preceding cases. The red tourmalines from Brazil and from Rumford, Maine, are very nearly represented by the expression—

$$Al_5(SiO_4)_6(BO_2)_2.BO_3R'H.Al_3R'H_2;$$

with Li : Na approximately as 5 : 4. The slight deficiency in the alkalis is made up by the presence of small amounts of calcium, iron, and manganese, but the ratio $Al_8 : Si_6$ is very clear. The green tourmalines are all lower in alumina, and range downward toward the iron end of the series; and like the latter are representable as mixtures of the following molecular types:—

* *Bulletin of the United States Geological Survey*, No. 167.

A. $Al_5(SiO_4)6(BO_2)_2.BO_3(AlOH).Fe_4H_4$.
B. $Al_5(SiO_4)6(BO_2)_2.BO_3Ca\,Fe_4H_4$.
C. $Al_5(SiO_4)6(BO_2)_2.BO_3LiH.Al_2Li_2H_4$.
D. $Al_5(SiO_4)6(BO_2)_2.BO_3NaH.Al_3NaH_2$.
E. $Al_5(SiO_4)6(BO_2)_2.BO_3H_2.Al_3NaH_2$.

Thus, the dark opaque green tourmaline from Rumford, Maine, is a molecular mixture corresponding to $A_6B_2C_7D_{14}E_5$; the similar mineral from Auburn is $A_8B_1C_8D_{17}$; the light green from Auburn $A_2C_3B_{10}D_8E_{12}$, and the nearly colourless from Auburn, $A_1B_5C_{20}D_{10}E_{35}$. From Brazil the dark green is $A_2B_1C_6D_8$, and the light green is $A_1B_2C_9D_8E_5$. The complexity of these expressions is only apparent, not real, as a study of the original type formulæ will show. They compare with the reduced analyses as follows:—

	Rumford.		Auburn, dark.		Auburn, medium.	
	Found.	Calculated.	Found.	Calculated.	Found.	Calculated.
SiO_2 ..	36 68	36·69	36·37	36·45	37·95	37·52
B_2O_3 ..	10·26	10·69	9·98	10·63	10·57	10·94
Al_2O_3 ..	38·26	37 74	36·89	37·21	38·11	39 02
FeO ..	6·78	6·90	7·94	7·72	4·47	4·28
CaO ..	0·34	0 34	0 17	0·16	0·50	0·50
Na_2O ..	3·12	3·07	3·19	3 14	2·58	2·58
Li_2O ..	0·95	0·94	1 06	1·07	1 35	1·35
H_2O ..	3·61	3·63	4·40	3·62	4·47	3·81
	100·00	100·00	100·00	100·00	100·00	100·00

	Auburn, pale.		Brazil, dark.		Brazil, light.	
	Found.	Calculated.	Found.	Calculated.	Found.	Calculated.
SiO_2 ..	38·25	37 92	37·24	37·14	37 49	37·64
B_2O_3 ..	10·30	11·05	9·95	10·83	10·32	10·97
Al_2O_3 ..	39 92	40·17	38 66	38·07	39·86	39·04
FeO ..	2·75	2·56	5·49	5·24	3·74	3·61
CaO ..	0·43	0·49	0·39	0·35	0·49	0·46
Na_2O ..	2·55	2·53	2 90	3·01	2 59	2 76
Li_2O ..	1·34	1 34	1·63	1·64	1·71	1 69
H_2O ..	4·46	3·94	3 74	3·72	3·80	3·83
	100·00	100 00	100·00	100 00	100 00	100 00

(To be continued).

ON TRIVALENT CARBON.

(Third Paper).*

By M. GOMBERG.

(Concluded from p. 21).

Compound of the Unsaturated Hydrocarbon with Acetic Ether.

A reaction entirely analogous to the one described above takes place when acetic ether is substituted for absolute ether. A compound of the unsaturated hydrocarbon with acetic ether is at once formed when triphenylchlormethane, dissolved in the ester, is treated with zinc. It also produced when the metal is allowed to act upon the halogen compound dissolved in benzene, and to the concentrated solution acetic ether is added. The sample which furnished the results given below was prepared by the second method; it was washed fifteen times with acetic ester, and was thoroughly dried in an atmosphere of carbon dioxide.

0·3160 grm. substance gave 1·0180 grm. carbon dioxide and 0·2080 grm. water.

	Calculated for $2(C_6H_5)_3C+CH_3CO_2C_2H_5$.	Found.
Carbon	87·74	87·86
Hydrogen	6·69	7·37

* From the *Journ. Am. Chem. Soc.*, xxiii., No. 7. The first paper appeared in the *Journ. Am. Chem. Soc.*, xxii., 757; the second in *Am. Chem. Journ.*, xxv., 320.

The compound of the hydrocarbon with the ester is perfectly colourless when first prepared, but soon turns pale yellow. It is more stable than the corresponding ether compound, and can be kept much longer without discolouration. It can be obtained in large crystals, or in the form of a granular crystalline powder. On heating, it loses the ester.

1·1170 grms. substance lost at 100° C. 0·1575 grm.

	Calculated for $2(C_6H_5)_3C+CH_3CO_2C_2H_5$.	Found.
Loss	15·50	14·10

The escaping acetic ester was condensed and identified as such. The residue, after driving off the ester, looked like that obtained from the ether compound. It was amorphous, yellow, soluble in benzene and in carbon disulphide; it absorbed iodine, and gave the peroxide. An analysis gave the following results:—

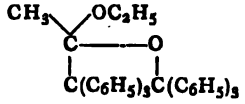
0·2135 grm. substance gave 0·7250 grm. carbon dioxide and 0·1322 grm. water.

	Calculated for $(C_6H_5)_3C$.	Found.
Carbon	93·76	92·61
Hydrogen	6·24	6·93

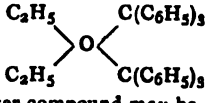
It will be noticed that in both instances the per cent of carbon is somewhat low for triphenylmethyl. This may be accounted for by a slight oxidation, but I have no explanation at present as to the cause of the rather high per cent of hydrogen.

What is the Nature of these Compounds?

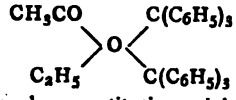
It is impossible to tell at this time whether in these two instances the ether and the acetic ester are those of crystallisation or of constitution. Such a distinction is at best but an arbitrary one. One would expect that ether of crystallisation would be easily driven off *in vacuo* over sulphuric acid—which is not the case in this instance. From the extreme unsaturation of the hydrocarbon it may be assumed that it might add itself to acetic ester, giving a body something like this:—



But it could hardly be expected that mere heating at 80—100° C. would break up a chain of three carbon atoms in such a way as to regenerate the ester. Then, again, the combination with ethyl ether precludes any such addition theory in that instance. If it is not ether of crystallisation, then the following constitution of that body suggests itself:—



and the acetic ester compound may be supposed to have the corresponding constitution:—



In support of such a constitution might be cited the remarkable avidity of the hydrocarbon for oxygen, and the possibility of oxygen to act as tetravalent. Such a possibility was foreshadowed by van't Hoff as long ago as 1877 ("Ansichten über die Organische Chemie"). There has since been accumulating a considerable amount of evidence of physical nature pointing to such a possibility. In connection with this may be mentioned the recent work of Kanonnikow. From a study of alcohols and ethers at their critical condition, Kanonnikow (*Journ. Russ. Phys. Chem. Soc.*, xxxiii., 197) comes to the conclusion that in

these compounds, under these conditions, the oxygen behaves as if it were tetravalent. The extreme unsaturation of triphenylmethyl presents an excellent opportunity to test the theory of the tetravalence of oxygen. The above formulæ for the two compounds must be looked upon, at present, as a mere suggestion. It is my intention to extend this study to other oxygen compounds, as well as to nitrogen derivatives, and I beg to reserve this field for further work.

ON PYRITE AND MARCASITE.*

By H. N. STOKES.

The object of this paper is to describe a chemical method by which the native forms of iron disulphide may be distinguished with certainty, and by which their relative amounts in mixtures of the two may be quantitatively determined. The method is applied to various supposed pyrites and marcasites, and it is shown that in the absence of well-marked crystallographic features it is extremely easy even for experts to be deceived as to the nature of a given specimen.

I. Uncertainty of Methods of Distinguishing Pyrite and Marcasite.

Pyrite and marcasite are usually distinguished by their differences of crystalline form, colour, density, and ease of oxidation.

Crystalline Form.

Pyrite usually crystallises in cubes, often in pentagon-dodecahedrons or octahedrons, and frequently in combinations of these and more complicated forms of the isometric system, while marcasite assumes various forms of the orthorhombic system. This criterion is inapplicable when the minerals assume the massive or compact form, or when they occur as concretionary nodules in which the crystalline form can not be made out. Moreover, pseudomorphs are not uncommon, and paramorphs of pyrite after marcasite and of marcasite after pyrite are said to occur, in which events no conclusion can be drawn from this source.

Colour.

The colour of pyrite is generally said to be pale brass yellow, while that of marcasite is described as tin-white, greyish white, greenish, brass-yellow, bronze-yellow, and in other terms. Dana† describes marcasite as pale bronze-yellow. These discrepancies are doubtless due to tarnished specimens having been described. Marcasite frequently assumes a yellowish tarnish, which may vary to copper coloured. On fresh surfaces, such as are obtained by breaking or by removing the film by warming with dilute hydrochloric acid, the colour is unquestionably tin-white or greyish white, closely matching platinum, and without a trace of yellow. The white colour is not an indication of the presence of arsenic (see *prox.*), as it is equally shown by specimens which are free from arsenic. To distinguish pyrite and marcasite by the colour, they must be cleaned with hydrochloric acid, and immediately examined under a good white light in comparison with standard specimens also freshly cleaned. The faint yellow of pyrite makes it extraordinarily easy to deceive oneself if the light be yellowish in tint. The differences of the supposed yellow and light-coloured pyrites vanish, except in rare cases, when these precautions are observed, and pyrite can usually be distinguished with ease when mixed with marcasite. In the case of fine-grained concretions, or where the surface is too rough to afford a good reflection, it is sometimes quite impossible to draw any conclusion from the colour.

* From the *Bulletin of the U.S. Geological Survey*, No. 186.

† "System of Mineralogy," 6th ed., p. 94; for a list of terms and references see Julien, *Annals New York Acad. Sci.*, 1887, vol. iv., p. 179.

Density.

Density according to Different Authorities.—The density of both minerals is subject to some variation, as shown by the following figures from different sources.—

Density of Marcasite and Pyrite.

Authority.	Marcasite.	Pyrite.
Dana (*a*)	4·85—4·90	4·95—5·10
Rammelsberg (*b*) ..	4·90	5·00
Julien (*c*)	4·80	5·01
Stokes (*d*)	4·88—4·90	5·02—5·04

(*a*) "System of Mineralogy," 6th Ed., Pp. 85, 94.
(*b*) *Zeit. Deutsch. Geol. Gesell.*, 1864, vol. xvi., p. 267. Supposed normal density.
(*c*) *Annals New York Acad. Sci.*, 1887, vol. iv., pp. 177, 210. Supposed normal density.
(*d*) See "Data for Pyrites" (*prox.*).

Density not a sure Criterion of Composition.—My own determinations were made with the pycnometer at 18—23°, on the purest obtainable material, coarsely powdered. Julien's figure for marcasite is certainly too low. The following list, giving the densities of some specimens of which I have determined the composition (after deducting impurities), shows how illusive may be conclusions as to the relative amounts of pyrite and marcasite based on density alone:—

Densities of some Specimens of Pyrite and Marcasite.

No. of specimen.	Density.	Composition, chemically determined.
5.	5·041	100 per cent pyrite.
3.	5·023	100 per cent pyrite.
33.	4·987	83·5 per cent pyrite, 16·5 per cent marcasite.
7.	4·891	100 per cent marcasite.
10.	4·886	100 per cent marcasite.
12.	4·880	100 per cent marcasite.
29.	4·856	99·5 per cent pyrite.
30.	4·843	99·5 per cent pyrite.
31.	4·819	100 per cent pyrite.
17.	4·563	100 per cent pyrite.

Oxidation.

While it is unquestionably true that marcasite oxidises more rapidly than pyrite under the same conditions, a compact, well-crystallised marcasite can be kept almost indefinitely without any change further than a superficial tarnish, whereas finely divided or porous pyrite oxidises with great rapidity, and this fact has frequently caused pyrite to be mistaken for marcasite.

The amount of oxidation depends upon the time during which the specimen has been exposed; upon the peculiar conditions under which it has occurred, such as exposure to dry or moist air, or to water holding oxygen or other oxidising agents in solution; upon whether the oxidation products are removed; upon the surface exposed per unit of volume; and upon an oxidation factor characteristic of each mineral, but probably not more than three times greater for marcasite than for pyrite. It is obvious, therefore, that no trustworthy conclusion can be drawn in any case from vitriolisation in the absence of definite knowledge of these factors; and even when the conditions of time and exposure are identical, as when the specimens are kept side by side in the cabinet, it is necessary to know the $\frac{\text{surface}}{\text{mass}}$ factor before rapid vitriolisation justifies one in pronouncing a specimen to be marcasite. One of the most extreme cases of rapid vitriolisation that I have seen is No. 21 (see *prox.*, "Quantitative Composition of Specimens of Doubtful Nature"), which consists of nodules of practically pure pyrite. Many of the rapidly vitriolising specimens consist of mixtures of pyrite and marcasite, and apart from the porosity existing in such cases it is possible that an electrochemical

action between the two may assist the oxidation. It is hardly necessary to add that the old idea that marcasite tends to produce ferrous sulphate, and pyrite to give limonite, is untenable.

Penfield's Method.

Penfield (Brush and Penfield, "Determinative Mineralogy," 15th Ed., p. 252) has described a method of distinguishing the two minerals, based on treating the finely powdered substance with strong nitric acid under identical conditions; pyrite dissolves completely, while marcasite leaves a residue of sulphur. While this procedure serves well to distinguish the pure minerals, it is obviously incompetent to detect pyrite in the presence of marcasite.

Action of Copper Sulphate on Pyrite and Marcasite.

A. P. Brown (*Proc. Amer. Philos. Soc.*, 1894, vol. xxxiii., p. 225; Chemical News, 1895, vol. lxxi., p. 179) asserts that when marcasite is heated for six hours at 200° with 10 per cent copper sulphate solution its iron is completely dissolved as ferrous sulphate, while under the same conditions pyrite yields a mixture of 2 mols. ferric and 1 mol. ferrous sulphate; and on this he bases the conclusion that in marcasite the iron is wholly in the ferrous condition, $Fe''S_2$, while in pyrite it is four-fifths ferric, $Fe''S_2(Fe'''S_2)_4$. As Brown's work might seem to afford a means of distinguishing the two minerals, and as the different constitution, if existing, would have an important bearing on the question of their formation in nature, I have repeated these experiments, but with totally different results. The details of my experiments are given further on, and I here state merely my conclusion that the only recognisable difference is the greater ease with which marcasite is attacked by the cupric solution, and that no evidence can be obtained in this way as to any difference of chemical constitution.

II. Behaviour of Pyrite and Marcasite toward Ferric Solutions.

In the course of an investigation on the action of ferric salts on natural sulphides, differences observed in the behaviour of pyrite and of marcasite were so marked as to make it seem probable that on these differences there might be based a method of distinguishing these minerals, and of determining them quantitatively in mixtures, thus establishing a sure means of ascertaining the composition of concretions and other doubtful specimens, and of testing the validity of the conclusion of Julien, quoted below (see *prox.*), that many supposedly pure pyrites and marcasites are mixtures of the two. While approximate results were soon reached, many fruitless experiments had to be made before all important sources of error were eliminated, and before the process could be brought to a reasonably satisfactory quantitative basis. Only the results obtained by the perfected method are presented in this paper.

Action of Ferric Chloride.

It has long been known that ferric salts attack pyrite. L. L. de Koninck (*Annales Soc. Geol. Belgique.*, 1883, vol. x., p. 101; *Zeit. Anorg. Chemie.*, 1901, vol. xxvi., p. 123) found that pyrite is oxidised at 170—200° C. by ferric chloride or ferric ammonium alum, with formation of a ferrous salt and sulphuric acid. J. H. L. Vogt (*Trans. Amer. Inst. Min. Eng.*, Richmond Meeting, February, 1901) mentions that pyrite is very slowly attacked in the cold by 30 per cent ferric chloride solution, giving a trace of sulphuric acid. He regards the formation of sulphuric acid as subordinate, and does not ascribe an important *rôle* to ferric salts in the decomposition of pyrite. Julien (*Annals New York Acad. Sci.*, 1887, vol. iv., p. 138—139) observed a reduction of ferric chloride by marcasite. No quantitative study of these reactions has been made, so far as I can ascertain.

Percentage of Sulphur Oxidised.

Preliminary experiments showed that very little dilute solutions of ferric salts attack pyrite rapidly at the boiling temperature, and much more slowly, but quite appreciably, in the cold. It is upon the determination of the percentage of sulphur oxidised in the total mineral decomposed that the new method is based. If ferric chloride be used, the amount of mineral decomposed can be found from the increment of iron in the solution, and the SO_4 determined as barium sulphate. An experiment with a ferric chloride solution containing 1·14 grms. Fe''' per litre, with a little hydrochloric acid, which was boiled to complete reduction with an excess of powdered pyrite free from sulphate, in a current of carbon dioxide, showed that 65 per cent of the sulphur was oxidised, a result agreeing well with the results presented below.

(To be continued).

NOTICES OF BOOKS.

Studies from the Chemical Laboratory of the Sheffield Scientific School. Edited by Horace L. Wells. New York: Charles Scribner's Sons. London: Edward Arnold. 1901. 2 vols. 8vo. Pp. 379 and 444.

The half-title page preceding the one given above bears the additional words: "Yale Bi-Centennial Publications"; and a note states that these volumes form part of a series prepared by the Professors and Instructors with the approval of the President and Fellows of Yale University, issued in connection with the Bi-Centennial Anniversary (held in the autumn of 1901), as a partial indication of the character of the studies in which the University teachers are engaged.

These sumptuously made and carefully edited volumes show what the Sheffield Chemical Laboratory has done and is doing in the way of scientific research, and the papers here reprinted from periodicals are limited to the work done by the present officials chiefly during the last ten years. These restrictions as to persons and time prevent the "studies" from giving an adequate conception of the vast amount of first-class investigations in chemistry that have been issued from the department of Yale University, of which the Sheffield Scientific School is so prominent a member. At the same time, these limitations enable the authorities to show to the public the activity of the present teaching forces, which comprise no less than eight professors and instructors in divers branches of chemistry.

No review of the researches contained in these volumes could be made satisfactory without some notice of the institution itself. The chemical department of the school was the earliest established; it was first conducted in what had been the "President's House," but in 1860, through the liberality of Joseph E. Sheffield, it was united to an Engineering Department (already in operation), and removed to Sheffield Hall, in which new and commodious laboratories were then constructed; there it remained for thirty-five years. The teaching force was gradually increased, a wider range of instruction was introduced, and the names of certain gentlemen associated with the school became widely known; notably, George J. Brush, the mineralogist; Samuel W. Jonson, the authority on agricultural chemistry; O. D. Allen, who had charge of analytical chemistry and metallurgy; and W. G. Mixter and Russell H. Chittenden, whose researches in physiological chemistry are renowned.

The first volume is devoted to papers on general inorganic chemistry and on double halogen salts; they are all reprints of articles that have appeared in the *American Journal of Science*, the *American Chemical Journal*, and elsewhere, and embrace a great variety of topics. "Cæsium Trihalides" form the subject of the first paper, by the editor of the volumes; then follow several other articles embodying further researches

on cæsium salts by the same author and his associates. Also one on the purification of cæsium material, for this rare element has herein received much attention. Other papers are on special analytical methods, on an isomer of potassium ferricyanide, on trivalent vanadium, &c.; only one dealing with points in pure theory, the Periodic System, by James Locke.

In the second half of Volume I. are papers on the double halides of several elements, among which cæsium and its comrades are prominent.

Volume II. contains numerous papers on organic chemistry, to which the name of Professor H. L. Wheeler is most frequently attached. Among them may be named those bearing the following titles;—"Researches on the Iso-Anilides," "On Diacid Anilides" (several papers), "Researches on the Cyclo-amidines, and on the Cyclo-Amides," "On the Re-arrangement of Imido-Esters," &c., all being contributions to knowledge of high rank.

Accompanying these is a bibliography of the principal publications of the present officers of the Sheffield Scientific School, so far as the work has been done in New Haven.

The "Studies" reflect great credit on their authors and on the institution whence they spring; the series of volumes of which these form a part make a notable exhibit of the activity of the famous Yale University. H.C.B.

Chemistry and Physics; a Manual for Students and Practitioners. By Walton Martin, Ph.B., M.D., and William H. Rockwell, Jun., M.D. Series edited by Bern B. Gallaudet, M.D. Illustrated with 137 Engravings. London: Henry Kimpton. 1901. Pp. 374.

Though not very clearly stated in the title, we gather that this book is intended specially for medical students and practitioners, to whom a knowledge of chemistry and physics is important. The authors have endeavoured as far as possible not to give undue prominence to the medical aspect of certain chemicals which are also drugs, nor to certain uses of electricity which are employed in the treatment of disease, as they feared to encroach upon Materia Medica and Electro-therapeutics. At the same time, those compounds, especially in the section of organic chemistry which are of medical interest, have received more detailed descriptions than those which are of interest only to the chemist pure and simple. The book is well and clearly printed and bound, and is furnished with a very complete index.

Die Normalelemente und ihre Anwendung in der Elektrischen Messtechnik. By Dr. W. Jaeger. Halle-a-S.: Wilhelm Knapp. 1902.

The recent developments in the theory of normal elements are dealt with at considerable length and with conspicuous clearness in this book. The Latimer Clark cell and its various modifications are dwelt upon very minutely both from a theoretical and practical point of view. In the section on theory Cohen's latest work on the thermodynamics of the normal element is well summarised, and in all respects the book seems comprehensive and well up-to-date.

Beiträge zur Chemischen Physiologie und Pathologie. I. Band. Hefte 1—4. Braunschweig: Friedrich Viewig und Sohn. 1901.

This new periodical, which is to be issued at short intervals under the editorship of Dr. Franz Hofmeister, is intended to meet the demand for a journal of biochemistry, occupying itself with pathology and physiology regarded from a chemical point of view, and treated by chemical methods. Judging from these two first issues, which contain much interesting matter communicated by well-known physiological chemists, the new publication seems likely to prove useful in extending our knowledge of the chemistry of the animal organs, secretions, &c. Special mention must be made of the reports of Dr. Leo Langstein's work on ovoglobulin and ovalbumin, and of Dr. H. Conradi's paper on the coagulation of the blood.

Roscoe-Schorlemmer's Ausführliches Lehrbuch der Chemie. By J. W. Brühl. *Organische Chemie.* Teil VII. Braunschweig: Friedrich Viewig und Sohn, 1901.

This volume brings to an end the complete revised edition of Roscoe and Schorlemmer's "Text-book of Chemistry," and includes the systematic and alphabetical indexes of the whole work so that they may be readily detached and bound separately.

The albuminous substances form the subject of the first and greater part of the book; following Hammarsten's classification, they are dealt with under the headings, (1) products derived from true albumens including albuminates, (2) true albumens, (3) proteids, and (4) albumenoids. The rest of the book is occupied with the bile substances, enzymes, and ptomaines; in the case of this last section, and indeed throughout the whole volume, much of the material has been collected from original papers and brought together for the first time in the form of a text-book, many of the researches described being of quite recent date.

This last volume fully maintains the high level of excellence reached in the previous volumes both in matter and arrangement, and it is much to be regretted that no English translation of the whole text-book is to be obtained. The world of science owes a debt of gratitude to those who produce a treatise such as this, embracing the whole range of pure chemistry, no less than to those who discover or enquire into new developments of the science.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxiii., No. 25, December 16, 1901.

This number contains no matter of chemical interest.

No. 26, December 23, 1901.

Dilution Constants of Saline Solutions.—Albert Colson.—The author experiments on the relation between the degree of dilution of a saline solution and the heat of solution. Solutions of NaCl, KCl, $NaNO_3$, and KNO_3 were tried, and a table of numbers is given.

Metallic Strontium and its Hydride.—M. Guntz.—The author obtained a considerable quantity of strontium by the electrolysis of an aqueous solution of its chloride, using a kathode of mercury. An amalgam is formed from which the strontium can be obtained by slow heating in a vacuum in an iron crucible, the mercury gradually distilling off. The properties of strontium are similar to those of barium. Strontium seems, however, to combine less easily with liquid ammonia.

Two Blue Oxides of Molybdenum.—G. Bailhache.—In a preceding paper the author described the preparation of blue oxide of molybdenum from the compound $Mo_2O_5.2SO_3$. A concentrated solution of $Mo_2O_5.2SO_3$ when exposed to the air gradually changes from brown to green, and finally to blue. At the same time a blue substance is deposited at the bottom of the vessel. The composition of this latter body is very variable. The blue solution when treated with ammonium chloride precipitates a blue oxide, but its purification is very difficult. The author now proves that this variation in composition is due to the formation of two oxides of molybdenum,

both blue in colour. These are obtained separately from $BaMoO_4$, and have the respective formulæ $Mo_2O_3.2MoO_4.6H_2O$ and $(Mo_2O_3)_3(Mo_7O_{24})_2.18H_2O$. These two oxides present absolutely the same characteristics. The only reaction by which they may be distinguished is their action on alkalis; a slightly different coloured precipitate is formed in the two cases. The author intends to further investigate this matter.

Chlorobenzoate and Dibenzoate of Methylene.—Marcel Descudé.—Chlorobenzoate and dibenzoate of methylene may be obtained simultaneously and in nearly equal proportions by the action of benzoyl chloride on trioxymethylene in presence of zinc chloride. The chlorobenzoate is a colourless liquid, heavier than water (sp. gr. = 1·236), in which it is insoluble. It solidifies at −15°, and is very highly refracting. It can be transformed quantitatively into the dibenzoate by heating equivalent weights of the chlorobenzoate and dry potassium benzoate for about four hours at a temperature of about 150°.

$$C_6H_5-\underset{Cl}{COO}>CH_2+C_6H_5-COOK=$$
$$=KCl+\underset{C_6H_5COO}{C_6H_5COO}>CH_2.$$

Hyposulphites of Aromatic Amines.—A. Wahl.—When a solution of sodium hyposulphite is added to a solution of aniline chlorohydrate, a voluminous crystalline precipitate is formed. These crystals, when re-dissolved in water at 30—40°, deposit on cooling, and when thus purified possess the composition of aniline hyposulphite $(C_6H_5NH_2)_2.H_2S_2O_3$. All the primary amines which the author examined, both in the benzenic series and in the naphthalenic series (aniline, ortho-, meta-, and paratoluidine, α- and β-naphthylamine), give normal hyposulphites corresponding to the formula $(RNH_2)_2H_2S_2O_3$. The secondary and tertiary amines do not give the corresponding hyposulphites.

New Reactions of Organometallic Derivatives (IV.). Synthesis of Ketones.—E. E. Blaise.—In a preceding paper the author showed that the ethero-organomagnesium derivatives react on the nitriles, and give in certain cases easily isolated crystalline compounds. These compounds are instantly decomposed by water, and give rise to ketones according to the following reaction:—

$$2\left[\frac{R}{R'}>C=N-Mg-I(C_2H_5)_2O\right]+4H_2O=$$
$$=\frac{R}{R'}>CO+Mg(OH)_2+MgI_2+2(C_2H_5)_2O.$$

This reaction is very general. It takes place in the cyclic series as in the fatty series, but the best yields are obtained with alcoholic iodides and cyclic nitriles.

Basic Properties and Tetravalence of Oxygen in the Xanthene Series.—R. Fosse.—The author first establishes the fact that the mono-halogenated derivatives of the xanthene series possess basic properties. He also shows that such bodies obtained either by the action of the halogens on the xanthenes or by the hydracids on the corresponding hydrols, behave in the same manner as amine salts by forming compounds with many reactions of alkaloids, &c.; in fact, the compounds formed seemed to behave like hypochlorites of these bases.

MISCELLANEOUS.

A Volumetric Method for the Estimation of Phosphorous Acid.—O. Kühling.—The solution is first oxidised with warm permanganate; it becomes alkaline and MnO_2 is deposited—

$$3PO_3H_3+2MnO_4K+H_2O=2PO_4H_3+2MnO_2+2KOH.$$

In order to get a good deposit of MnO_2 it is advisable to add sulphate of zinc, which reacts on the potash. The following are the details of the method. The liquid containing from 0·32 to 0·7 grm. of PO_3H_3 in 20 or 40 c.c. is poured into an Erlenmeyer flask of 250 c.c. capacity, treated with 20 to 40 c.c. of a 10 per cent solution of $ZnSO_4+7H_2O$, then with a not quite sufficient quantity of $KMnO_4$, heated on the water-bath until the colour disappears, then again treated with more $KMnO_4$, heated again, and finally treated with the small amount of $KMnO_4$ which is absolutely necessary for the colouration to persist after heating for fifteen minutes. The solution is filtered again and an excess of $KMnO_4$ added, then heated on the water-bath with frequent stirring; on cooling it is diluted with an equal volume of water, and the precipitated MnO_2 collected on an asbestos filter, and after careful washing it is placed in a flask and treated with an acidulated solution of potassic iodide. The oxide is dissolved in the cold by the help of frequent shaking; this sets free the iodine which is titrated by hyposulphite. As an alternative the excess of $KMnO_4$ in the filtrate can be determined direct.—*Berichte*, vol. xxxiii., p. 2914.

Action of Peroxide of Hydrogen on the Hyposulphites.—A. Nabl.—On adding together equimolecular portions of peroxide of hydrogen free from H_2SO_4 and a solution of hyposulphite of soda, the author could not detect the formation of a hydrosulphite as announced by MM. Bernthsen and Bazlen, but he observed that the liquid became sensibly warmer and took an alkaline reaction. Equal molecules of sulphate and sulphite of sodium were formed, as well as a basic substance oxidisable by an excess of H_2O_2 and not very stable, as the liquid when left to itself soon becomes cloudy and gives off SH_2. An attempt was made to isolate this sulphurised base by acidulating with hydrochloric acid after the action of the H_2O_2 was completed, then evaporating to dryness on the water-bath, taking up with water, filtering, and precipitating with $HgCl_2$. A partial reduction of the salt takes place; the base gives a double mercuroso-mercuric salt, which, after washing, was treated with SH_2. The filtered solution was evaporated almost to dryness on the water-bath, and, on the addition of alcohol and dilute sulphuric acid, a fine floculent white precipitate of a sulphate was formed. This last salt was collected on a filter, washed with etherised alcohol, re-dissolved in water, then treated with carbonate of baryta, when it gave a colourless liquid with a green fluorescence and having a strongly alkaline reaction; the return is not good. This substance precipitates the metallic oxides from their saline solutions, reduces Fehling's solution slowly when warmed, and with $SnCl_4$ gives stannous sulphide. Its chloroplatinate becomes reduced slowly and spontaneously in the cold, but rapidly when warmed. It is probable that this base contains the radical SOH and can be compared with Cahours' hydrate of trimethylsulphine, or again with hydroxylamine.—*Berichte*, vol. xxxiii., p. 3093.

MEETINGS FOR THE WEEK.

MONDAY, 20th.—Society of Arts, 8. (Cantor Lectures). "The Purification and Sterilisation of Water," by Samuel Rideal, D.Sc., F.I.C.

TUESDAY, 21st.—Royal Institution, 3. "The Cell—its means of Offence and Defence—Immunity," by Allen Macfadyen, M.D., B.Sc.

—— Society of Arts, 8. "The Architect's Use of Enamelled Tiles," by Halsey Ricardo.

WEDNESDAY, 22nd.—Society of Arts, 8. "Scientific Observations at High Altitudes," by Rev. J. M. Bacon.

THURSDAY, 23rd.—Royal Institution, 3. "Recent Excavations at Delphi and in the Greek Islands," by A. S. Murray, LL.D., F.S.A.

—— Society of Arts, 4.30. "Bengal—The Land and its People," by F. H. Skrine.

FRIDAY, 24th.—Royal Institution, 9. "The Discovery of the Future," by H. G. Wells, B.Sc

—— Physical, 5. "The Factors of Heat," by James Swinburne. Exhibition of some Twinned Crystals of Selenite, by Eustace Large.

SATURDAY, 25th.—Royal Institution, 3. "Landmarks in the History of Opera" (with Musical Illustrations), by W. H. Hadow, M.A., Mus.Bac.

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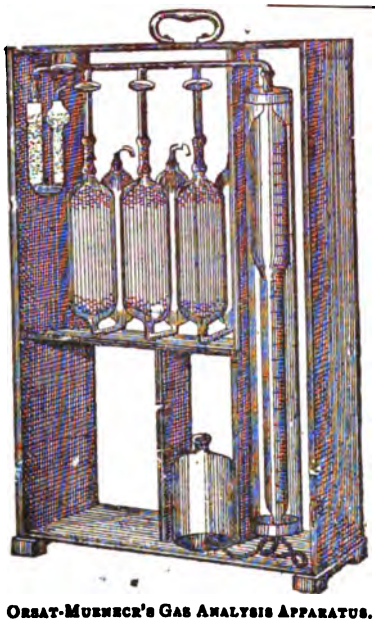

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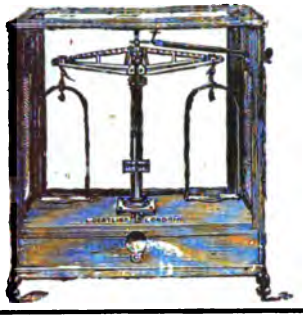

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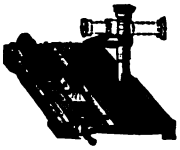

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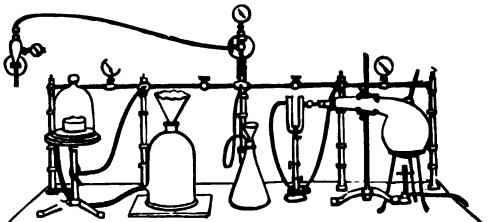

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RESEARCHES ON THE PRESSURE FORCES OF LIGHT.

By PETER LEBEDEW.

In the exposition of his electromagnetic theory of light, Maxwell (1873) took into consideration those forces which appear as forces capable of doing work ("weight moving") in an electrically or magnetically polarised medium. As a necessary consequence of his theory it follows that these forces must appear in a pencil of rays, and Maxwell says:—

"In a medium in which an undulation is propagated a pressure acts in the direction of propagation, and this pressure is at every spot numerically as great as the energy referred to unit volume at that spot."

Heaviside, Lorentz, Cohn, and Goldhammer have recently more closely investigated Maxwell's deduction concerning the existence of such pressure forces.

In quite a different way, and apparently without knowing of Maxwell's result, Bartoli (1876) arrived at the same conclusion; he describes a series of processes by which it is said to be possible to transfer by means of movable mirrors the radiating energy from a colder to a warmer body, and calculates the work to be done according to the second main proposition. The necessity for the performance of work in moving the mirror in the direction opposite to that of the incident ray forces us to the assumption of pressure forces which are exerted by this ray on the mirror. Bartoli calculated the magnitude of these pressure forces; his result agrees perfectly with that obtained by Maxwell.

The method employed by Bartoli was followed by Boltzmann, and afterwards by Prince Galitzin and Guillaume in calculating the pressure forces which are exercised by the ray; Drude extended this method to absolutely black bodies.

If a beam of parallel rays falls normally on a plane surface, the magnitude of this pressure of Maxwell and Bartoli, p, is determined if the quantity of energy per unit of time, E, the reflecting power of the surface, ρ, and the velocity of transmission of the ray, V, are known. We then have—

$$p = \frac{E}{V}(1+\rho);$$

where ρ lies between 0 for an absolutely black substance and 1 for a perfectly reflecting substance.

These pressure forces are very small. Maxwell as well as Bartoli has calculated that the rays of the sun falling normally on 1 sq. c.m. exert a pressure which amounts to 0·4 m.grm. for an absolutely black surface, and 0·8 m.grm. for a plane mirror.

Conjectures that such pressure forces of the rays must exist have been expressed long before this time. So Kepler (1619) believed that the explanation of the force of repulsion which the sun exercises on comets' tails was to be sought for in the pressure forces of its rays, an idea which agreed with the prevailing emission theory of light, and which was ardently defended by Longomontanus. The same reason lead Euler (1746) also to ascribe pressure forces to the sun's rays, and he tried to establish the necessity for the existence of such forces on the movements of light considered as longitudinal vibrations.

De Mairan (1754) with Du Fay made very interesting attempts to prove these suppositions experimentally; but he soon preceived that the warming of the surrounding air prevented any sure proof of the existence in light of forces capable of doing work. Considering the means at his disposal in the eighteenth century, De Mairan's experiments deserve the greatest admiration. A. Fresnel (1825) also made similar attempts, and likewise met with the same difficulties; an investigator of similar phenomena led Crookes to the discovery of the radiometer forces. The applications of which these pressure forces of Maxwell and Bartoli are capable, both in physics and astronomy, render an experimental investigation of them specially desirable, as both Maxwell and Bartoli's theoretical deductions rest upon certain simple properties of the absorbing and reflecting surfaces; and it would appear doubtful whether from these properties alone the forces capable of doing work could be determined for *light* rays also. A direct experiment leads most simply to this end.

Zöllner and Bartoli's efforts in this direction have led to no positive result; for this reason I undertook the following experimental research on the pressure forces of light:—

I. *The Preliminary Experiments.*

In his text-book Maxwell says:—"Concentrated electric light will probably exert a still greater pressure (than sun rays), and it is not impossible that the rays of such a light if they fell on a thin metallic leaf delicately suspended in a vacuum would produce an appreciable mechanical effect."

When I wished to begin my experiments the order of procedure proposed by Maxwell seemed to be hopeless, because already Zöllner had followed this method without result, and also "had observed that the value (of light pressure) obtained theoretically by Maxwell was about 100,000 times smaller than the value of the results observed by Crookes* in a special case." Even if one could reckon on appreciably diminishing these disturbing radiometer effects, I thought nevertheless, that only such contrivances by which these radiometer forces were compensated could give the desired result.

In research on the radiometer forces Schuster had proved that these forces are intrinsic forces of the radiometer. Righi proved this in an elegant way. He says:—"I let a radiometer float in water head downwards, so that the glass cap of the mill lay on the tube which kept the latter from falling out of the normal position by means of which a friction resulted, which stopped the revolution of the mill. When an intense beam of rays was directed on the mill, not the least revolution (of the radiometer) occurred." Bertin and Garbe obtained the same result on the repetition of this experiment.

I applied Righi's method of procedure to the determination of Maxwell and Bartoli's pressure forces of light as follows:—Between two circular covers stamped out of very thin nickel leaf a piece of mica was bent to the form of a cylinder, and securely fastened—consequently, a cylindrical radiometer body was formed—in which the vane was rigidly fastened to the covering. This radiometer was hung in an exhausted glass globe by a glass thread. When the light of an arc lamp was directed on the vane immediately revolution of the radiometer was observed,† the magnitude of which corresponded to Maxwell and Bartoli's pressure forces.

In the experiments when, for the sake of comparison, I investigated the effect on the radiometer vane by itself *without* the mica covering, I found that the radiometer forces which appeared were considerably smaller than those mentioned by Zöllner, and even produced an

* In his calculation Zöllner took the energy of total radiation of a candle too small. If we compare the radiometer forces observed by E. Nichols (*Wied. Ann.*, 1897, lx., p. 405) with the Maxwell and Bartoli press forces calculated from the radiation measurements of K. Angström (*Wied. Ann.*, 1899, lxvii., p. 647) we get a proportion of about 10,000.

† Righi, as well as Bertin and Garbe, observed no effect of the ressure forces of Maxwell and Bartoli in their experiments *only* ecause their methods intended for the detection of the very much reater radiometer forces were not sensitive enough for the forces of ght.

essentially smaller disturbance, like the disturbances resulting on the proportionally great radiometer covering,

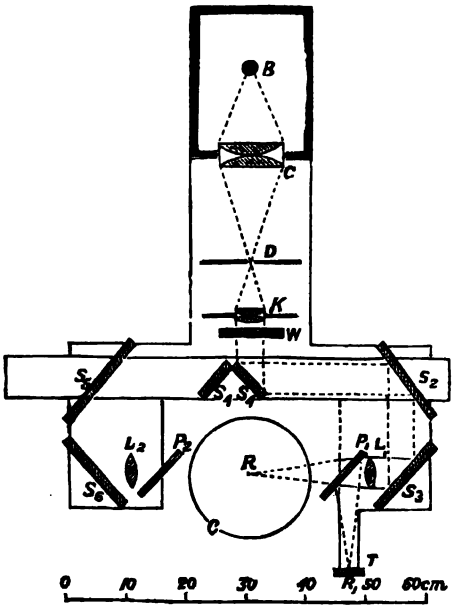


FIG. 1.

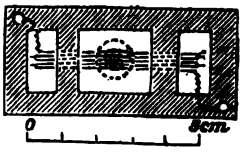


FIG. 2.

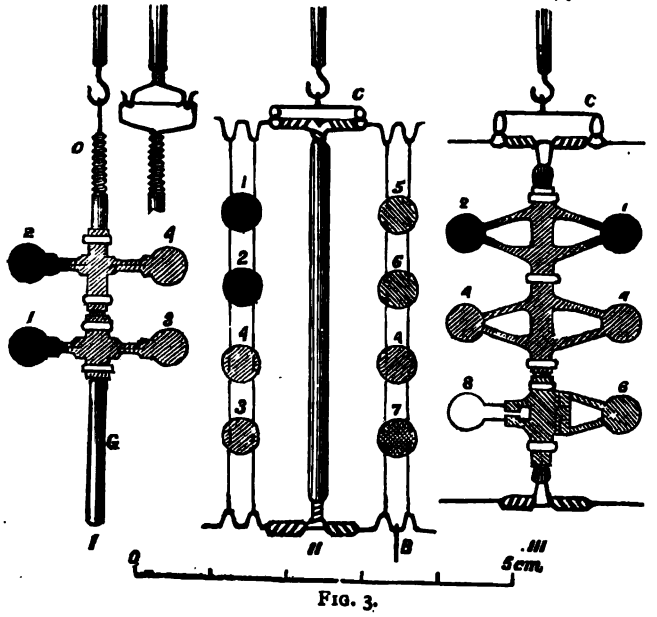


FIG. 3.

owing to convection. Therefore, I gave up this method, and undertook the research in the simple way proposed by Maxwell.

II. *The Apparatus and Preparation of the Experiment.*

Two difficulties are met with in carrying out research on Maxwell and Bartoli's pressure forces of light; the disturbances caused by convection and the radiometer forces which arise. By the greatest rarefaction these disturbing forces are diminished, but must, nevertheless, be taken into consideration in the measurements.

The disturbances by convection are due to the fact that on warming the vane by exposing it to the light the surrounding atmosphere is warmed also, and a slowly ascending current is caused. If the plane of the vane has even a small (almost negligible) inclination to the vertical plane, the rising current of gas exercises a force of torsion on the vane which is dependent *only* on the degree of warming, but *not* on the direction in which the heating rays fall. This disturbance can be eliminated by directing the beams from the same source, first on one side and then on the other side of the vane.

The disturbance by radiometer forces were reduced to a minimum by making the glass globe as large as possible, excluding by means of a ray-filter all rays which could be absorbed by the glass wall of the globe, making the vanes of thin metal leaf in order to equalise the distribution of heat, and, moreover, exhausting the globe as completely as possible by means of a mercury pump and the application of freezing mixtures.

If the radiometer forces are small, the corresponding correction can be calculated as follows:—The radiometer forces are directly proportional to the difference of temperature between the illuminated and dark surfaces of the vane, or for two vanes of the same material having their surfaces in the same state proportional to their thicknesses.* If we make simultaneous observations of *two* similar vanes of very different thicknesses we can *calculate* how far the light would cause the vane to turn, if the thickness of the vane were zero, and also the radiometer force zero. I must here remark that this correction is to be made only in the case of platinised vanes; for metal vanes the radiometer forces were, contrary to all expectation, indefinitely small.

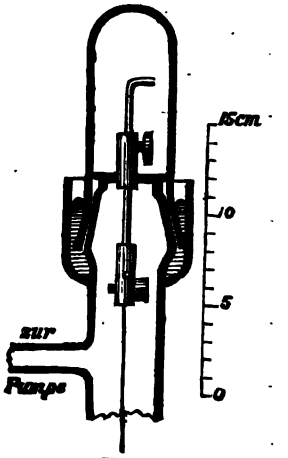


FIG. 4.

* In our experiments the temperature difference between the illuminated vane and the globe was very much greater than the temperature difference between both surfaces of the vane. To whichever function of the first temperature difference the radiometer forces may be proportional, their "weight moving" effect on the vane shows their difference to both surfaces of the vane, and this is approximately directly proportional to the second temperature difference.

Besides the well known disturbing influences just mentioned another hypothesis must not remain unnoticed, namely, that the dispersion of light by illuminated surfaces, pointed out by Lenard and Wolf, could cause perceptible "weight moving" reaction forces, and these must always appear as an accompaniment of Maxwell and Bartoli's pressure forces of light; but these hypothetical reaction forces must be dependent on the colour of the incident light as well as on the chemical nature of the vane. Experiments with coloured light and with different vanes have not led to the discovery of any appreciable effects of these reaction forces.

The following arrangement was used:—The image of the terminal of a direct current arc lamp, B (30 ampères), was cast on a metal diaphragm, D (d = 4 m.m.), by means of a condenser, C. The cone of light coming from the diaphragm was converted into a beam of parallel rays by the lens, K. In order to free the light from ultra-red rays, behind the lens, K, was a glass vessel filled with pure water,* parallel to the plane of the lens (thickness 1 c.m.); in the place of this a ruby-red glass could be used, the pure water being replaced by an ammoniacal solution of copper.†

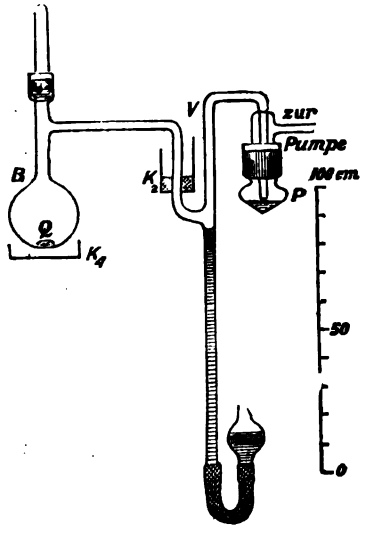


Fig. 5.

On its further passage the pencil of rays was reflected from the plane glass mirrors, S_1, S_2, and S_3, situated in the rear, and then re-united by the lens L_1 to a real enlarged (d' = 10 m.m) image at R of the diaphragm D in the interior of the glass globe. If the pair of mirrors, S_1 and S_4, were moved along, the rays would pass similarly, and fall on the vane suspended in the glass globe G from the other side. The lenses L_1 and L_2 had a focal length of 20 c.m., and a diameter of 5 c m., so that the incident light has an aperture angle of about 15°.

The mirror apparatus was rigidly connected with the arc lamp. The lamp stood on a sliding base on which it could be turned towards or away from the glass globe. The cone of light could be raised or lowered by means of an adjusting screw.

The sudden fluctuations in the intensity of the light, which are unavoidable in the case of an arc lamp, can be rendered harmless only by making many observations.

In order to be able to reduce the separate series of measurements to a constant mean light-intensity, the following arrangement was used:—Between the lens L_1 (Fig. 1) and the glass globe G a thin plane glass plate, P_1, is placed at an angle of 45° to the direction of the rays. The greater number of the rays pass through the glass plate without hindrance, while the reflected light combines to form a real image, R_1, and this falls on the thermopile T. The thermopile (Fig. 2) is composed of five Constantin iron elements (wire thickness, d = 0·025 m.m.), which are mounted in an ebonite frame and closed with glass plates; the relative light-intensity is measured by the deflections of a d'Arsonval's galvanometer. In order to diminish similarly the light coming from the lens L_2 (Fig. 1), a corresponding glass plate, P_2, is interpolated. The light-intensity can only be controlled if the two mirrors, S_1, S_4 (Fig. 1), stand in the positions shown in the diagram. If they are moved, no light falls on the thermopile, and this position determines the galvanometer zero point.

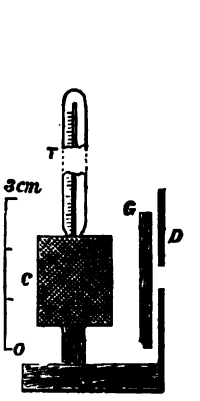


Fig. 6.

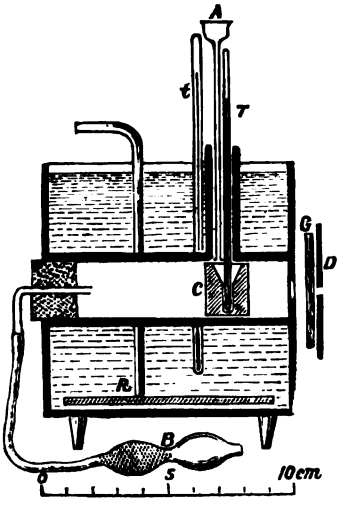


Fig. 7.

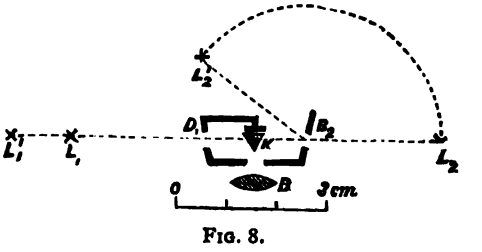


Fig. 8.

Three different kinds of vane apparatus were used (Fig. 3).

Vane apparatus I. consists of a glass rod, G, to which two cross-pieces made of platinum-leaf of different thicknesses were rigidly fixed by means of platinum wire rings (without cement); in order to make the circular discs (diameter 5 m.m.) of the same size, a steel stamp was used. Two of the discs were blank on both sides; two others platinised on both sides—the thicker five times more strongly than the thinner. To suspend the apparatus on the double hook of a torsion arrangement, a platinum loop, O, melted into the glass rod G was used; this was perpendicular to the direction of the vane arms, so that on suspension the glass rod G was free to move vertically in the plane of these arms.

Vane apparatus II. (Fig. 3, II.) consisted of a glass rod into which arms of platinum wire were melted. Between these platinum arms fine platinum wires (0·05 m.m.) were

* Thus all ultra-red rays with $\lambda > 1{\cdot}2\ \mu$ are excluded; glass lenses stop the ultra-violet rays.

† The available energy is reduced by the red and the blue ray filters to about one-fifth that of white light; that is a proof that the radiation employed belongs almost exclusively to the visible part.

stretched, which went through little holes in four metal discs, and held these firmly in a vertical position in the plane of the platinum arms; these platinum wires were so thin that their radiometer effect could be neglected. The vane apparatus was provided with a Cardani's suspension arrangement of platinum wire, C, and by means of this hung on the platinum hook of the torsion thread; the vertical position of the glass rod G was secured by means of an additional weight of platinum wire, B.

Vane apparatus III. was similarly constructed to I., and had a Cardani's suspension arrangement. The strips of tin plate which served as arms for the discs were very narrow (0·3 m.m.), and yet they satisfactorily ensured the vertical position of the discs.

The mica wing (8) was fastened in an aluminium setting. Aluminium arms were placed at the top and bottom to prevent the vanes striking against the glass wall of the neck of the globe while the apparatus was being suspended.

The following vanes were experimented with:—

No.	Material.
1.	Platinum, thickly platinised.
2.	,, five times more thickly platinised.
3.	,, blank. Thickness = 0·10 m.m.
4.	,, ,, ,, = 0·02 ,,
5.	Aluminium, ,, ,, = 0·10 ,,
6.	,, ,, ,, = 0·02 ,,
7.	Nickel, ,, ,, = 0·02 ,,
8.	Mica. ,, < 0·01 ,,

As a torsion apparatus, a glass thread (length 30 c.m.) was used. At its lower end it carried a plane mirror, and above it was fastened in an *iron* clamp in the interior of the arrangement of Fig. 4 in the neck of the globe. In order to fasten the torsion thread *without cement*, its ends were secured between two pieces of ignited asbestos cardboard and fastened above by means of the clamp, and below by a platinum ring to the mirror support.

The mirror was set in a thin aluminium setting, blackened with platinum chloride, and was platinised by deposition at the cathode, as even a covered silver mirror soon becomes destroyed by mercury fumes. Wellmann-Martin's method of illuminating the scale has proved most useful in overcoming the feeble reflection of such a mirror and the effects of the interposition of the arched wall of the glass globe.

For the determination of the direction force from observations of the oscillations, the torsion thread was removed from the globe, the vane apparatus taken away, and the thread loaded with a copper-wire 4 c.m. long and having a mass of 0·314 grm.

The observations were made with three different torsion threads, whose direction forces were so chosen that if the scale distance were 1200 divisions the blank vanes gave a double deflection of 40 to 90 divisions. The three kinds of vane apparatus were found to have oscillation periods of fifteen, thirty-five, thirteen seconds respectively.

A Kahlbaum's pump was used to exhaust the globe. The measurements with a McLeod and Kahlbaum's manometer gave as result that a partial atmospheric pressure of 0·0001 m.m. (scarcely a fifteenth of mercury vapour pressure at the temperature of the room) could easily be obtained.

To obtain a still higher degree of exhaustion, the following method was employed:—Some mercury, Q, was poured into the globe B, the pump was started, and the mercury was warmed about 5° C. above the temperature of the room by means of a water-bath, K, placed under B; the mercury distils into the pump, which works continuously and draws with it the air in the globe B. If, by raising the mercury in the barometer tube, V, the globe is shut off from the pump and the dry vessel, P, only mercury vapour remains behind in the globe. By the usual method of cooling with ice and salt in K_1 and K_2 the tension of the mercury vapour can be made very small.

The quantity of energy of radiation incident on the vane was measured by a calorimeter. The lamp with the mirror apparatus (Fig. 1) was drawn so far back on the sliding base that the vane apparatus could be replaced by a diaphragm, D (Fig. 6 or Fig. 7), which was exactly the same size as each vane (d=5 m.m.); all rays which passed through the diaphragm D were absorbed by a calorimeter. In these measurements, a glass disc, G, compensated for the reflection from the glass wall of the globe; it was interposed between the diaphragm and the calorimeter in order to stop the radiation of heat from the diaphragm.

The first calorimeter (Fig. 6) consisted of a copper block, C; the vertical hollow of this is filled with mercury, and in it is placed the bulb of a small thermometer, T, graduated in ½° C.; the absorbing surface was blackened. The total water equivalent of the calorimeter was 3·13 grms. (the specific heat of copper taken as 0·093).

The second calorimeter (Fig. 7) was a copper cylinder of similar construction with a water equivalent of 3·61 grms.; its absorbing surface was gilded and then platinised; it was placed in a brass vessel in a water-bath of about 1 litre capacity, which was provided with a stirrer, R.

In order to cool the calorimeter below the temperature of the water-bath before beginning the experiment, a few drops of ethyl ether were poured by means of the glass tube, A, into the conical opening of the calorimeter, and a strong current of air drawn through the brass vessel by means of indiarubber bellows, B.

The measurements gave as a result that the quantity of energy of radiation incident on the diaphragm (d=5 m.m.) amounts to 1·2 to 1·8 grm.-cals. per minute, *i.e.*, that this intensity amounts to from twice to three times the total radiation of the sun on the earth's surface.

A Ritchie's photometer serves to measure the reflection of the metal leaf employed (Fig. 8). The light of two small incandescent lamps, L_1 and L_2, passed through the diaphragms, D_1 and D_2 (diameter 3 m.m.), and fell on a little prism of chalk, K, whose edges are observed through a magnifying glass, B. The lamp L_1 was adjusted till the brightness of the two lamps appeared equal; then L_2 was turned through an angle of about 130° towards L'_2, the diaphragm D_2 was closed from without by the metal leaf under examination, and again L_1 adjusted to L'_1, where its brightness equalled that of L'_2. For the angle of incidence of 25°, the reflecting power, Q, of the metal leaf is given by—

$$Q = (KL : KL'_1)^2.$$

(To be continued).

ON THE RELATION OF THE VISCOSITY OF MIXTURES OF SOLUTIONS OF CERTAIN SALTS TO THEIR STATE OF IONISATION.*

By JAMES BARNES, B.A., Dalhousie College, Halifax, N.S.

(Concluded from p. 31).

Mixtures of Solutions.

As there is no change of volume on mixing the constituent solutions of the above electrolytes of the concentrations given below (see *Trans. N.S. Inst. Sci.*, 1895-96, ix., 125; also 1897-98, ix., 297, and 310), and as the solutions mixed were of equal volume and also equimolecular, the expression (2) for the value of a property in the case of a mixture of two electrolytes with a common ion, reduces to:—

$$P = P_w + \frac{n}{2}[k_1(1-a_1)+l_1a_1+k_2(1-a_2)+l_2a_2] \quad . \quad . \quad (3).$$

where n is the concentration of the solutions, and the k's and l's have the values obtained above for simple solutions

* *Transactions of the Nova Scotian Institute of Science*, x., p. 113.

of the respective electrolytes. For the application of this equation to the calculation of the viscosity of a mixture, all the quantities required are known except the a's, the ionisation coefficients in the mixture.

Determination of Ionisation Coefficients in the Mixture.

The method proposed by Professor MacGregor (*Trans. N.S. Inst. Sci.*, 1895-96, ix., 101; also 1898-99, x., 68) for finding the ionisation coefficients in a mixture of two electrolytes having an ion in common, is by solving graphically the following equations:—

$$\frac{a_1}{V_1} = \frac{a_2}{V_2} \quad \ldots \quad (4),$$

$$N_1V_1 + N_2V_2 = 1 \quad \ldots \quad (5),$$

$$\frac{a_1}{V_1} = \int_1(V_1) \quad \ldots \quad (6),$$

$$\frac{a_2}{V_2} = \int_2(V_2) \quad \ldots \quad (7),$$

where the electrolytes are denoted by 1 and 2, the concentrations (in grm.-equivalents per litre) of the mixture with respect to them by N_1 and N_2 respectively, their ionisation coefficients by a_1 and a_2, and their regional dilutions (in litres per grm.-equivalent) by V_1 and V_2, the regional dilutions being the dilutions of the electrolytes in the regions which they are supposed to occupy in the mixture, or the dilutions of the constituent isohydric solutions.

His graphical mode of solving these equations involves the drawing of dilution-ionic-concentration curves, which, as they have great curvature for moderately dilute solutions, cannot be drawn with great accuracy unless a large number of observations of the conductivity are available. As mentioned above, extensive series of observations of the conductivity in the case of the salts under consideration were available; but they were all made at 18° C., and required therefore to be reduced to 25° C. before they could be used. In order to reduce this labour as much as possible I devised another mode of solution which requires only a comparatively small number of observations. It is based on the fact that the specific-conductivity-concentration curve of an electrolyte exhibits only slight curvature, and can therefore be drawn with fair accuracy from a small number of observations.

The above equations may be expressed in terms of specific conductivity and concentration as follows:—Since

$$\frac{a_1}{V_1} = \frac{\mu_1}{V_1\mu_{\infty 1}} = \frac{k_1}{\mu_{\infty 1}} \quad \ldots \quad (8),$$

and—

$$\frac{a_2}{V_2} = \frac{k_2}{\mu_{\infty 2}} \quad \ldots \quad (9),$$

where k_1 and k_2 are the specific conductivities of the electrolytes in the regions which they respectively occupy in the mixture, and the μ_∞'s the specific molecular conductivities at infinite dilution for each electrolyte, equation (4) becomes:—

$$\frac{k_1}{\mu_{\infty 1}} = \frac{k_2}{\mu_{\infty 2}} \text{ or } k_1 = \frac{\mu_{\infty 1}}{\mu_{\infty 2}} k_2 \quad \ldots \quad (10).$$

From equation (5) we obtain:—

$$\frac{N_1}{C_1} + \frac{N_2}{C_2} = 1 \quad \ldots \quad (11),$$

where C_1 and C_2 are the regional concentrations. Equations (6) and (7) are based on the fact that at a definite temperature the conductivity is a function of the concentration alone. They therefore take the following forms:—

$$k_1 = f_1(C_1) \quad \ldots \quad (12),$$

and—

$$k_2 = f_2(C_2) \quad \ldots \quad (13).$$

There are thus four equations (10—13) for the determination of the four unknown quantities:—k_1, k_2, C_1, and C_2.

These equations can be solved graphically. Equation (12) is employed by drawing a curve having as abscissæ the values of the specific conductivities and corresponding values of the concentrations as ordinates. Before equation (13) is used the values of the conductivities are multiplied by the constant $\frac{\mu_{\infty 1}}{\mu_{\infty 2}}$. Then these new values are plotted against the corresponding concentrations, on the same coordinate paper, to the same scale as employed for equation (12). From these two curves one finds by inspection two points, one on each curve, having a common abscissa, according to equation (10), and ordinates (C_1 and C_2) such that by substituting their values in equation (11) it will be satisfied. These points can be found after two or three trials. Thus one has determined k_1, C_1, and C_2; k_2 being found by multiplying k_1 by the constant $\frac{\mu_{\infty 2}}{\mu_{\infty 1}}$. The a's are now obtained from equations (8) and (9); for the reciprocals of the C's give the V's.

Results of the Calculations on Mixtures.

The following Table VI. contains the requisite data for the calculation, by formula (3), of the viscosity of mixtures of solutions of the salts under consideration; and it shows the agreement of the values thus calculated with the observed values. The ionisation coefficients of the salts in the mixture are determined as above, and the concentrations are expressed as in the former tables.

TABLE VI.—*Viscosity at 25° (Kanitz).*

Concentration constituent solutions.		Ionisation coefficients in mixture.		Observed values.	Calculated values.	Difference.
KCl.	NaCl.	KCl.	NaCl.			
1·0	1·0	0·745	0·667	1·0390	1·0419	+0·0029
0·5	0·5	0·775	0·728	1·0180	1·0173	−0·0007
0·25	0·25	0·807	0·783	1·0070	1·0069	−0·0001
KCl.	½BaCl₂.	KCl.	½BaCl₂.			
1·0	1·0	0·756	0·552	1·0429	1·0533	+0·0101
0·5	0·5	0·779	0·613	1·0159	1·0220	+0·0061
0·25	0·25	0·811	0·675	1·0049	1·0082	+0·0033
½K₂SO₄.	½Na₂SO₄.	½K₂SO₄.	½Na₂SO₄.			
1·0	1·0	0·535	0·434	1·1660	1·1670	+0·0010
0·5	0·5	0·597	0·517	1·0773	1·0768	−0·0005
0·25	0·25	0·641	0·604	1·0334	1·0354	+0·0020
½K₂SO₄.	½CuSO₄.	½K₂SO₄.	½CuSO₄.			
1·0	1·0	0·559	0·152	1·2240	1·2423	+0·0183
0·5	0·5	0·612	0·210	1·1060	1·1107	+0·0047
0·25	0·25	0·668	0·256	1·0485	1·0510	+0·0025

Kanitz regards his observed values as affected by a possible error of ± 3 in the third decimal place. Considering the many calculations necessary to obtain the calculated values—first, in finding the specific molecular conductivity at infinite dilution for 25° C., and also ionisation coefficients at 25° from the data at 18°, and then in the determination of the ionisation coefficients of the salts in the mixture by the graphical method—the agreement between the observed and the calculated values (calculated, it should be noted, with the ionisation constants obtained for the simple solutions), is very satisfactory, especially in the case of solutions of potassium chloride and sodium chloride, and solutions of potassium sulphate and sodium sulphate, where the differences are all within the limit of experimental error. In the case of the stronger solutions of potassium chloride and barium chloride, and of potassium sulphate and copper sulphate, the differences are not within the limit of error; but a close agreement, as was pointed out in the beginning, could not be expected. It will be noticed, however, that the differences in these cases diminish and approach the experimental error as concentration diminishes. Observations on the viscosity of weaker solutions of these salts were not available.

From these results, therefore, it may be concluded, that

the viscosity of mixtures of dilute solutions of the salts under consideration can be predicted, by the aid of a dissociation theory, within the limit of experimental error, from data as to the viscosity and conductivity of the constituent solutions only.

ON PYRITE AND MARCASITE.*

By H. N. STOKES.

(Continued from p. 34).

II. Behaviour of Pyrite and Marcasite toward Ferric Solutions (*continued*).

Ferric Sulphate Oxidation Method.

Owing to the tediousness of the gravimetric methods and of the SO_4 determination in the presence of iron, I have adopted a much more expeditious indirect method, which involves the use of ferric ammonium alum and titration with potassium permanganate.

Principle of the Method.—The oxidation of FeS_2 by $Fe_2(SO_4)_3$ may be regarded as taking place by two independent reactions:—

(1) $FeS_2+Fe_2(SO_4)_3=3FeSO_4+2S$.

(2) $2S+6Fe_2(SO_4)_3+8H_2O=12FeSO_4+8H_2SO_4$.

If the reaction be carried out in the presence of an excess of FeS_2 to complete reduction of the ferric salt, it is easily seen that equation (1) holds even if the theory of Brown be true, that the iron in pyrite is partly ferric, for in this case the ferric salt formed will in turn be reduced by the excess of pyrite. Upon the extent to which the second reaction takes place, as compared with the first, depends the percentage of sulphur oxidised in that portion of the mineral which is decomposed.

If a ferric solution containing a known amount of iron be boiled with an indefinite excess of FeS_2 under appropriate precautions, and if the increment of iron in the solution be then determined, as well as the ferrous salt formed, this increment gives the amount of sulphide decomposed: three times this increment gives the quantity of ferrous salt formed in equation (1); while any excess of ferrous salt is that formed by oxidising sulphur to sulphuric acid as in equation (2). From these data the percentage of sulphur oxidised may be computed.

Deduction of an Expression for Oxidised Sulphur.

It is unnecessary to know the absolute quantities of iron and sulphur involved in these reactions, or the strength of the permanganate solution. It suffices, in deducing an expression for the oxidised sulphur, to employ symbols expressing the permanganate equivalent of the iron, and the symbols used represent simply the volumes of permanganate consumed by a given volume of the solution.

For a given volume let—

a = iron in the original solution,
b = resulting ferrous iron,
c = resulting total iron.

Then—

$c-a$ = increment of iron resulting from decomposition of FeS_2,

and—

$2\left(\frac{31{\cdot}83}{55{\cdot}60}\right)(c-a)$ = total sulphur in decomposed sulphide (A).

Also,—

$3\,(c-a)$ = ferrous iron produced according to equation (1).

and—

$b-3\,(c-a)$ = ferrous iron produced by oxidation of sulphur.

According to equation (2), 1 atom of sulphur requires for oxidation 6 atoms of ferric iron, producing 6 atoms of ferrous iron; hence,—

$$\frac{1}{6}\cdot\frac{31{\cdot}83}{55{\cdot}60}(b-3\,(c-a)) = \text{sulphur oxidised} \quad . \quad . \quad \text{(B)}.$$

Calling the percentage of sulphur oxidised p, we obtain from (A) and (B)—

$$p=\frac{\frac{100}{6}\cdot\frac{31{\cdot}83}{55{\cdot}60}(b-3\,(c-a))}{2\left(\frac{31{\cdot}83}{55{\cdot}60}\right)(c-a)}=\frac{8{\cdot}333\,b}{c-a}-25.$$

It thus appears that three titrations suffice to determine the percentage of sulphur oxidised, and that neither the amount of FeS_2, the volume of the ferric solution used, nor the absolute titre of the latter or of the permanganate need be known. As, however, the proportion of sulphur oxidised varies with the strength of the ferric solution, the extent of reduction, and the temperature, it is necessary, in order to obtain comparable results, to use a solution of standard composition and a standard temperature, and to continue the action to complete reduction.

Standard Solution and Temperature.

The standard solution contains 1 grm. ferric iron per litre, and is prepared by dissolving in water 8·61 grms. clear crystals of pure ferric ammonium alum, adding 16 c.m.³ 25 per cent sulphuric acid, and diluting to 1 litre. One hundred c.m.³ of this solution must show a decided change of colour with one drop of the permanganate solution, containing about 1·5 grms. per litre, and must be free from nitrate. The sulphuric acid is added to prevent precipitation of basic salt. The experiment is conducted at the boiling temperature. The strength of the ferric solution may vary 1 per cent without appreciable influence, and at least 99·5 per cent must be reduced.

Oxidation Coefficient.

The percentage of sulphur oxidised under these conditions, and with the precautions explained in detail below, may be called briefly *the oxidation coefficient* of the pyrite or marcasite. The oxidation coefficient of pyrite varies between 60 and 61, and that of marcasite between 16·5 and 18. The oxidation coefficients of mixtures of the two indicate the relative amount of each present, by reference to an empirical curve obtained with artificial mixtures of known composition. A theoretical deduction of the relative proportions from the oxidation coefficient is impossible in the absence of the necessary physical data.

Theory of the Reaction.

The mass law states that the active mass of a solid phase is constant; hence, the final composition of the resulting solution must be the same, irrespective of the relative amount of reagent and sulphide used, provided only that the latter, the solid phase, be always in excess. With more, or more finely powdered, material*—that is, with a relatively greater exposure of surface, equilibrium is sooner reached, but the composition of the resulting solution, and therefore the percentage of sulphur oxidised, is the same. It is unnecessary either to weigh the material or to measure the liquid accurately, but in order to make unavoidable experimental errors as nearly as possible constant, it is desirable always to employ approximately equal quantities, and to boil for nearly equal times.

Influence of Concentration.

Since the equation expressing the oxidation of the sulphur is of a higher order than that denoting the oxida-

* From the *Bulletin of the U.S. Geological Survey*, No. 186.

* Ostwald (*Zeit. Physik. Chemie*, 1900, vol. xxxiv., p. 495) has shown for certain cases that an extremely finely powdered substance is more soluble than the same in coarser particles, and that the composition of the resulting solution is therefore subject to slight variations. Such effects were looked for in the present instance by using coarse and very fine material obtained by sedimentation, but if they exist they are smaller than the errors of the method.

tion of the iron, it follows that the rate of oxidation of the sulphur must fall off more rapidly than that of the iron, with decreasing concentration of the ferric salt; hence, the value of p must constantly decrease as reduction proceeds, and comparable results can be obtained only by carrying the reduction to completion. This is shown in the following cases, where the experiment was stopped after partial reduction:—

Results of Experiments showing Constantly Decreasing Value of p *as Reduction Proceeds.*

Pyrite.		Marcasite.	
Fe reduced.	p.	Fe reduced.	p.
Per cent.	Per cent.	Per cent.	Per cent.
67·8	71·4	69 2	23·3
88·7	65·2	78·5	22·5
90·5	64·9	100·0	16·2
95·2	63·2		
99·5	61·5		
100·0	60·4		

Influence of Temperature.

The influence of temperature on p is shown by the following experiments at 20° and at boiling temperature, the reduction being complete in each case:—

Results of Experiments showing Influence of Temperature on p.

	Temperature.	p.
Pyrite	20°	80·8 per cent
	100°	60·4 ,,
Marcasite	20°	30 6 ,,
	100°	17·4 ,,

From the variation in the relative oxidation of the sulphur with concentration and temperature, it appears certain that no single equation expresses the oxidation of pyrite and marcasite by ferric salts to ferrous sulphate, sulphur, and sulphuric acid, but that it is the result of two, or possibly three, independent reactions of different orders, which produce results varying according to circumstances. It is therefore extremely probable that other oxidising agents, as atmospheric or dissolved oxygen, behave in a similar manner. The oxidation with separation of free sulphur presents no difficulties; as is well known, finely powdered pyrite and marcasite on exposure to the air for a short time give free sulphur, which can be detected by extraction with ether, and the formation of sulphur dioxide has been noted by various observers; finely divided moist marcasite evolves it strongly when heated on the water-bath. The difficulty is rather to account for the complete oxidation of the sulphur usually occurring in nature. Whether with stronger solutions and continued re-oxidation by air, or the presence of retarding agents, 100 per cent can be reached can not be stated at present, and experiments bearing on this point are desirable.

Reasons for the Different Behaviour of Pyrite and Marcasite.

The different proportion of sulphur oxidised under identical conditions in the two cases is doubtless due to the different solubilities of pyrite and marcasite, or at least the different rates at which they are attacked. That the sulphur is not completely oxidised is due to its smaller oxidation rate under the given conditions. The iron is attacked so fast that the oxidation of the sulphur falls behind, and a portion is liberated as free sulphur, which, as my experiments show, is scarcely oxidised by the boiling ferric solution when collected to aggregates of sensible size. This phenomenon is a familiar one when precipitated sulphides are treated with nitric acid; a portion of the sulphur is oxidised, and the remainder is liberated. When a ferric salt is reduced by hydrogen sulphide the sulphur of the latter is partly liberated, but a very considerable part is oxidised to sulphuric acid.* Whatever retards the solution of the sulphide without affecting the concentration of the oxidiser gives more time for the complete oxidation of the sulphur. This may be brought about by certain salts in solution or by a difference of physical structure in the crystals. Pyrite dissolves more slowly than marcasite, hence its sulphur is more completely oxidised. Under conditions where the solution is very slow, it may perhaps approximate completeness in both cases, a subject well worthy of investigation from its bearing on the oxidation of pyrites in nature.

Further Applications of the Method.

In general, when a mineral is composed of two or more elements, each capable of oxidation, but at different rates, there will be certain conditions under which one will be oxidised to a greater extent than the other, and the amount of this difference may often be determined by analysis of the solution. If the compound is dimorphous, one form will be more soluble than the other, and this will show a greater difference in the degree of oxidation of the two constituents than the less soluble form. The above method therefore makes it possible to distinguish not only between dimorphous sulphides but other dimorphous compounds, such as arsenides, and to determine their relative amounts in mixtures. It should also be possible to determine whether certain complex minerals are mixtures of two distinct kinds of molecules, or whether all the components are combined in one molecule. FeAsS, for example, should show a behaviour different from that of a mixture FeS_2 and $FeAs_2$ in equimolecular proportions. When the metal is more slowly oxidised than the non-metallic constituent a separation of a portion of the former may be expected. Gold sulphide, for example, might give sulphuric acid and free gold. As pointed out below, it is possible to ascertain in certain cases the form of combination of constituents which exist only as impurities; in a 3 per cent cupriferous pyrite, chalcopyrite can be distinguished from chalcocite and bornite, a determination which could not be made by analysis.

(To be continued).

* "Gmelin Kraut," vol. i., Part 2, p. 219. I have found that when a 1 per cent boiling solution of ferric chloride is reduced by hydrogen sulphide very nearly one-third of the reduction is effected by the sulphur.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING DECEMBER 31ST, 1901.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To CHARLES PERRIN, Esq., M.Inst.C.E.,
Water Examiner, Metropolis Water Act, 1871.

London, January 10th, 1902.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 192 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from Dec. 1st to Dec. 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 192 samples examined by us during the month, all were found to be clear, bright, and well filtered.

TABLE A.—*Rainfall in Inches at Oxford, Month by Month, during the Year* 1901.

	Actual fall.	Mean of 35 years.	Difference from the mean.	
	Inches.	Inches.	Inch.	Inch.
January ..	1·04	2·08	−1·04	—
February ..	1·31	1·80	−0·49	—
March ..	1·44	1·47	−0·03	—
April	1·96	1·61	—	+0·35
May	1·24	1·75	−0·51	—
June	1·52	2·10	−0·58	—
July	4·66	2·49	—	+2·17
August ..	2·12	2·38	−0·26	—
September..	1·86	2·39	−0·53	—
October ..	1·18	2·76	−1·58	—
November ..	0·54	2·30	−1·76	—
December ..	3·29	2·16	—	+1·13
	22·16	25·29	−6·78	+3·65

For the third time only during this year we have to record an excess of rain. The rainfall measured at Oxford during December was 3·29 inches, the average is 2·16 inches; this gives an excess of 1·13 inches, and reduces the previous deficit to 3·13 inches, or 12·3 per cent for the whole year, on the thirty-five years' average, as will be seen by Table A.

Our bacteriological examinations of 494 samples have given the results recorded in the following table; we have also examined 37 other samples, from special standpipes, wells, &c., making a total of 531 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 24 samples) ..	387
New River, filtered (mean of 120 samples) ..	18
Thames, unfiltered (mean of 24 samples) ..	4065
Thames-derived water, from the clear-water wells of eight Thames-derived supplies (mean of 270 samples)..	31
Ditto ditto .. .. highest	324
Ditto ditto .. .. lowest	0
River Lea, unfiltered (mean of 24 samples) ..	446
River Lea, from the East London Company's clear-water wells (mean of 32 samples) ..	14

Of the 422 samples of the impounded and filtered water supplied to London and examined bacteriologically during

TABLE B.—*Table showing Average Monthly Bacterial Variations during the Year* 1901.

Month.	Thames, unfiltered.	Thames-derived Companies, filtered.	New River, unfiltered.	New River, filtered.	River Lea, unfiltered.	River Lea (East London), filtered.	
January	4069	49	671	28	450	16	
February	5310	26	800	5	172	18	
March..	2303	27	593	10	315	43	
April	2076	15	176	6	177	15	
May	852	9	106	6	209	13	
June	1371	26	204	8	488	56	
	2663	25	425	10	302	27	Mean of 1st 6 months.
July	5560	35	383	13	837	40	
August	1646	22	284	13	223	16	
September	873	19	215	7	266	17	
October	865	13	173	4	155	12	
November	1909	25	280	10	202	13	
December	4065	31	387	18	446	14	
	2486	24	287	10	355	18	Mean of 2nd 6 months.
	2575	24	356	10	328	22	Mean of 12 months.

TABLE C.—*Average Number of Microbes in the Filtered London Waters for the Past Five Years.*

	Thames-derived Companies.	New River.	River Lea.
1897	46	32	62
1898	34	30	25
1899	27	15	20
1900	21	9	10
1901	24	10	22

TABLE D.—1901.—*Averages of the Supplies derived from the River Thames.*

	Common salt per gallon.	Nitric acid per gallon.	Hardness. Degrees.	Oxygen required per gallon.	Organic carbon per gallon.	Organic carbon per gallon.	Colour. Brown : Blue.
	Means.	Means.	Means.	Means.	Means.	Maxima.	Means.
January	2·194	1·047	16·12	0·058	0·161	0·253	21·7 : 20
February	2·189	1·101	16·46	0·053	0·148	0·185	17·4 : 20
March	2·227	1·035	15·64	0·056	0·172	0·237	18·5 : 20
April	2·193	0·796	15·20	0·068	0·177	0·234	19·4 : 20
May	2·109	0·776	14·68	0·042	0·106	0·139	11·6 : 20
June	2·112	0·711	14·12	0·040	0·090	0·137	10·1 : 20
July	2·160	0·717	13·28	0·039	0·075	0·100	9·5 : 20
August	2·047	0·634	12·85	0·040	0·076	0·090	9·6 : 20
September ..	2·243	0·639	12·75	0·035	0·080	0·094	7·2 : 20
October	2·335	0·631	13·83	0·036	0·088	0·103	6·7 : 20
November	2·406	0·940	15·19	0·035	0·094	0·115	8·8 : 20
December	2·506	0·991	15·43	0·043	0·104	0·152	13·4 : 20

the past month, 21 samples, or 4·9 per cent, were sterile. Eleven samples contained more than 150 microbes, while twenty-seven samples, or 6·3 per cent, contained over 100 microbes per c.c. The mean number of microbes in the twenty-seven excess samples was 185, against a corresponding mean of 154 in fourteen excess samples during November.

During the past year we have examined 6893 samples of London waters bacteriologically, as compared with 6731, 4792, and 3500 respectively in the three previous years. We have also made 2522 chemical analyses of London waters throughout this period, making a total of 9415 samples examined during the year.

The results of the filtration of the London waters are given in Tables B, C, and D.

Table B shows that during the first six months the Thames-derived Companies' clear-water wells contained on the average 25 bacteria per c.c., while the New River and River Lea supplies contained respectively 10 and 27. During the second six months, the number of bacteria in the waters from the three sources were respectively 24, 10, and 18.

An examination of the accompanying tables of the average composition, chemical and bacteriological, of the London water supply, proves that the quality on the whole was excellent, and that the high standard of previous years was well maintained during 1901. The care and attention which has been given to the general character of the supply is seen from the large number of samples—nearly 10,000—examined during the year.

It is remarkable that the rainfall in the Thames Valley was 12·3 per cent below the thirty-five years' average, a deficit substantially the same as that which occurred in the year 1900. We have had now seven yearly periods in which a deficiency of rain has occurred in the Thames Valley, as measured at Oxford, the aggregate deficit amounting to no less than 25·31 inches. Under these exceptional circumstances it is worthy of remark that London has suffered no restriction in the matter of supply, although many towns in the country have experienced considerable inconvenience from having to curtail their consumption of water.

We are, Sir,
Your obedient Servants,
William Crookes.
James Dewar.

THE CONSTITUTION OF TOURMALINE.*

By F. W. CLARKE.

(Concluded from p. 32).

In these cases the low boric acid of the analyses and the uncertainties as to the significance of the water determinations account for the chief variations between observation and theory. There is another complication also, due to the fact that alternative expressions are possible between which it is very difficult to decide. In the tourmaline from Haddam Neck, Connecticut, analysed by Penfield and Foote, a somewhat different commingling of molecules seems to be necessary, partly on account of the higher proportion of lime in the mineral and partly on account of the fluorine. This tourmaline also admits of various alternatives in formulation, but it agrees well with the molecular mixture—

3. $Al_5(SiO_4)_6(BO_3)_2.BO_3Ca.Fe_4H_4$,
10. $Al_5(SiO_4)_6(BO_3)_2.BO_3(AlOH).Al_2Li_2H_4$,
1. $Al_5(SiO_4)_6(BO_3)_2.BO_3Ca.Al_3NaH_2$,
6. $Al_5(SiO_4)_6(BO_3)_2.BO_3NaH.Al_3NaH_2$;

in which Ca is equivalent to a replacement of NaH. This mixture, with the group AlOH proportional to fluorine, gives a good comparison between analysis and theory, thus—

	Found.	Reduced.	Calculated.
SiO_2	36·96	36·75	36·86
B_2O_3	11·00	10·94	10·74
TiO_2	0·03		
Al_2O_3	39·56	39·35	39·44
FeO	2·14	4·35	4·43
MnO	2·00		
MgO	0·15		
CaO	1·28	1·27	1·15
Na_2O	2·10	2·09	2·07
Li_2O	1·64	1·63	1·54
H_2O	3·10	3·62	3·77
F	1·13		
	101·09	100·00	100·00

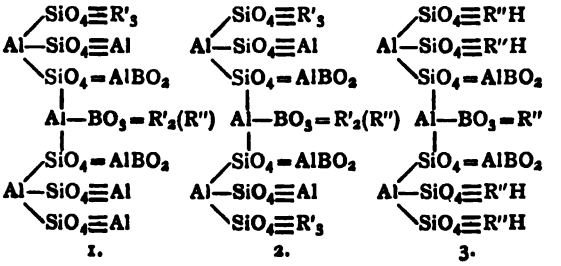
The theoretical amount of fluorine needed to replace hydroxyl in the assumed group, AlOH, is 0·97 per cent. Altogether, the comparison is fairly satisfactory.

One analysis by Riggs, that of the magnesium tourmaline from Hamburg, New Jersey, I have omitted from my discussion. In that tourmaline there are variations which I cannot readily account for, unless by assuming the presence in it of a molecule—

$$Al_5(SiO_4)6(BO_2)_2.BO_3R'_2.R''6.$$

Such a molecule, written structurally, exhibits affinities to garnet rather than to the micas; and I prefer to await further evidence before committing myself to any definite formulation in this instance. As for the analyses published by Jannasch and Kalb, they fit in well with those of Riggs, and are amenable to the same treatment.

At first glance, some of the formulæ which I have proposed may seem to be complex; but they are all of the same type, and can be reduced to a few general expressions, as follows:—



These formulæ cover all of the established variations in the composition of tourmaline; they render the various replacements or isomorphous admixtures intelligible, and they indicate the directions into which the species commonly alters. There is one objection to them, namely, that one of the end-products contains no alkali metal, and no alkali-free tourmaline is known. The same objection applies to the Penfield-Foote formula, as will be seen by anyone who attempts to apply it in the discussion of the iron tourmalines. Under either system of formulation the existence in tourmaline of alkali-free salts must be assumed.

One further possible advantage in the proposed formulæ remains to be pointed out. All of the chemists who of late years have discussed the composition of tourmaline agree in adopting the ratio between silicon and boron of 2 : 1, or $4SiO_2 : B_2O_3$. And yet many of the analyses vary from this ratio to an extent which may not be due to experimental errors. For example, from among Riggs's analyses the following cases show large variations, the boron being too low. I give the silica and boric oxide as determined, the boric oxide as calculated from the silica by the accepted ratio, and the amount of variation between the two.

* *Bulletin of the United States Geological Survey*, No. 167.

	SiO_2.	B_2O_3 found.	B_2O_3 calculated.	Difference.
Rumford, red ..	38·07	9·99	11·10	−1·11
Paris, black . ..	35·03	9·02	10·22	−1·20
Monroe, brown ..	36·41	9·65	10·62	−0·97
Brazil, green ..	36·91	9·87	10·76	−0·89
Auburn, colourless	38·14	10·25	11·12	−0·87

In the analyses by Jannasch and Kalb the following examples are very striking:—

	SiO_2.	B_2O_3 found.	B_2O_3 calculated.	Difference.
Snarum	35·64	9·93	10·40	−1·47
Mursinka	34·88	8·94	10·17	−1·23
Buckworth	35·50	8·34	10·35	−2·01
Brazil	37·05	9·09	10·81	−1·72

It would hardly be wise to dismiss these variations as due to errors, especially when the summation of the analyses is good and the analysts are known to be trustworthy. Such errors on the part of either Jannasch or Riggs would be almost incredible, and I am therefore inclined to believe that the analyses are good, and that we should seek a cause for the variations. In my scheme of formulation the bi-valent group of atoms $=Al—BO_2$ occurs. Replace this in part by the similar groups $=Al—OH$ and $=Al—F$, and the variations are accounted for. This supposition satisfies the analyses completely, and covers the ground. It is in accord with all the evidence, even though its validity may not be definitely proved. By its application to the discussion of the analyses, the divergences between the calculated composition and the composition as found can be notably diminished.

But although the formulæ which I have adopted serve to express the composition of all tourmalines, they still leave room for alternatives. Penfield and Foote, as well as myself, assume that tourmaline is a mixed salt containing distinct boric and silicic radicles. Future investigation may prove that it is really derived from a complex boro-silicic acid, as yet unknown; and the same conception may be true of other species, such as axinite, datolite, danburite, cappelenite, &c. A series of boro-silicic acids is theoretically conceivable; and until this question has been considered, the constitution of all the minerals above mentioned must be regarded as unsettled.

EXPERIMENTS RELATIVE TO THE CONSTITUTION OF PECTOLITE, PYROPHYLLITE, CALAMINE, AND ANALCITE.*

By F. W. CLARKE and GEORGE STEIGER.

In a series of investigations by Clarke and Schneider, which were carried out in the laboratory of the United States Geological Survey between 1889 and 1892 (*Bull. U.S. Geol. Survey*, No. 78, p. 11; *Ibid.*, No. 90, p. 11; *Ibid.*, No. 113, p. 27), a number of reactions were examined which shed some light upon the constitution of several natural silicates. The work then begun was unfortunately interrupted for several years; but it is now resumed, with the hope that it may be pushed considerably further.

Two of the reactions studied by Clarke and Schneider were of peculiar interest. First, in the case of talc, it was found that one-fourth of the silica could be liberated by ignition; and that the fraction thus set free was measurable by solution in aqueous sodium carbonate. This reaction suggests that other acid metasilicates may behave in a similar way, and that we perhaps have a means of discrimination between such salts and other compounds which simulate them. In other words, an acid metasilicate may be experimentally distinguished from a pseudo-metasilicate by the way in which it splits up when ignited. Evidence bearing upon this supposition will be found in the present paper.

The second of the reactions just referred to is that between dry ammonium chloride, at its temperature of dissociation, and various silicates (*Bull. U.S. Geol. Survey*, No. 113, p. 34). This involves, in part at least, the action of dry gaseous hydrochloric acid upon the compounds which are studied; and different minerals are very differently attacked. Some are almost completely decomposed, others are affected but slightly; and here again there seems to be a method of diagnosis which deserves further attention. Both reactions suggest the main purpose of the investigation; which is, the fractional analysis of silicates by means of various reagents, in order to gain evidence bearing upon their chemical structure. The evidence, at least, is of value, whether the interpretation of it be right or wrong. Each fact helps to the ultimate solution of the central problem, the problem of constitution.

Pectolite.

The pectolite which was chosen for examination was the well-known radiated variety from Bergen Hill, New Jersey. The mineral was in long, white needles, and apparently quite pure; but the analysis shows that it contained some carbonate as an impurity. Enough of the material was ground up to furnish a uniform sample for the entire series of experiments, and the work properly began with a complete analysis. The results obtained are as follows:—

Analysis.

SiO_2	53·34
Al_2O_3	0·33
CaO	33·23
MnO	0·45
Na_2O	9·11
Total H_2O	2·97
CO_2	0·67
	100·10

Fractional Water.

At 105°	0·27
At 180°	0·16
At 300°	0·22
At redness	2·32
	2·97

All of the water was given off at a barely visible red heat; and the figures show that practically all of it is constitutional, a fact which perhaps hardly needed re-verification. The analysis gives the accepted formula for pectolite, $HNaCa_2Si_3O_9$. Does this represent, as is commonly assumed, a true metasilicate? If it does, we should expect that ignition would split off silica proportional to the acid hydrogen, or one-sixth of the total amount. To answer this question several portions of the pectolite were sharply ignited, to complete dehydration, and then boiled each for fifteen minutes with a solution of sodium carbonate containing 250 grms. to the litre. In the extract so obtained the silica was determined; and three experiments gave the following percentages:—

8·96
8·67
8·42

Mean, 8·68

One-sixth of the total silica is 8·89 per cent; and the experiments, therefore, justify the original expectation. The belief that pectolite is a metasilicate is effectively confirmed.

Upon the unignited pectolite the sodium carbonate solution has a slow decomposing action, both silica and bases being withdrawn. In two experiments fifteen minutes of boiling extracted 2·07 and 2·55 per cent of silica, and by a treatment lasting four days 4·80 per cent

* *Bulletin of the United States Geological Survey*, No. 167.

was taken out. With water alone similar results were obtained; the action being so rapid, although relatively slight, that pectolite, moistened, gives an immediate and deep colouration with phenolphthalein. By boiling the powdered pectolite with distilled water alone 1·65 per cent of silica was brought into solution, and the ignited mineral, similarly treated for fifteen minutes, gave 1·78 per cent. The extraction in these cases is really an extraction of alkaline silicate, as the two following experiments prove. In A the unignited pectolite was boiled for fourteen hours with distilled water; and in B the mineral after ignition was subjected to like treatment for four hours. The dissolved matter in each case was determined, with the subjoined results:—

	A.	B.
SiO_2	2·98	3·03
CaO	0·30	0·10
Na_2O	0·81	1·50
	4·09	4 63

In A, no simple ratio appears; but in B the extracted silicate approximates very nearly to the salt $Na_2Si_2O_5$ In each instance the ratios vary widely from those of the original mineral, showing that actual decomposition and not a solution of the pectolite as such has occurred.

In the experiments upon pectolite the heating with dry ammonium chloride was omitted, for the data are already given in the original paper by Schneider and Clarke. In their experiments the mineral was thrice heated with ten times its weight of the reagent to above 350°, and then leached out with water. In the solution 20·50 per cent of lime and 6·95 of soda were found, with part of the manganese; showing that a very considerable decomposition had taken place. Possibly, by repeated treatments with ammonium chloride a complete decomposition might be effected, but this question is one upon which it seemed unnecessary to spend further time.

(To be continued).

CORRESPONDENCE.

ON THE MUTUAL ACTION OF ALUMINA AND FERRIC OXIDE AT INCIPIENT WHITE HEAT.

To the Editor of the Chemical News.

Sir,—In reference to the article on the above subject (Chemical News, lxxxiv., 305), and Mr. Riggs's letter on the combination of ferric oxide and zinc oxide (Chemical News, lxxxv., 24), it may be of interest to state that apparently zinc oxide also combines with aluminium oxide when a mixture of the two oxides is exposed to a full red heat.

For instance, when the precipitate thrown down by ammonia and ammonium sulphide in a solution containing zinc and aluminium is ignited sharply in a muffle, and then treated repeatedly with boiling concentrated hydrochloric acid, the alumina left insoluble retains considerable amounts of zinc oxide.

Similarly, by firing a heated mixture of metallic aluminium and excess of zinc oxide by means of a piece of magnesium ribbon, a dark grey mass is produced which shows no sign of fusion. On extracting this with hydrochloric acid a residue containing variable proportions of both zinc oxide and aluminium oxide is left behind. The composition of one substance obtained in this way, which appeared to be homogeneous under the microscope, was 86 00 per cent Al_2O_3 and 14·05 per cent ZoO.

The preparation of an actual compound, however, free from any excess of alumina, presents some difficulty.—I am, &c.,

Arthur G. Levy.

London, January 18, 1902.

THE INSTITUTE OF CHEMISTRY.

To the Editor of the Chemical News.

Sir,—I have addressed the enclosed to the President of the Institute of Chemistry.—I am, &c.,

A. Wynter Blyth.

To the President of the Council of the Institute of Chemistry.

Sir,—I have been solicited by more than one Fellow of the Institute to support their candidature as members of the Council. Those who have done so are I doubt not well fitted to be elected, but though I have been in my time on the governing body of many societies this kind of canvassing is entirely new to me, and appears so objectionable and so much against the best interests of the Institute that I earnestly ask the Council to take some means of suppressing it.

If the system is allowed to continue the vacancies that occur from time to time will not be filled by the most eminent, but by the most energetic—by those individuals who take the trouble to personally solicit the votes of the Fellows. The effect of this will be that the Council will cease to be respected.—I am, your obedient servant,

A. Wynter Blyth.

Town Hall, Marylebone Lane, W.
January 14, 1902.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxiii., No. 27, December 30, 1901.

Critical Constants and Molecular Complexity of Higher Hydrocarbons.—Ph. A. Guye and Ed. Mallet.—The authors make a series of new measurements of critical constants of higher hydrocarbons, choosing in most cases bodies containing chemical groups, different from those previously examined by other experimenters, and amongst these compounds, in all possible cases, choosing those having a high critical temperature.

Extension of Kirchhoff's two Laws.—E. Carvallo.—The author's researches lead him to enunciate the two following laws:—(1). The total flow of current across any closed surface is zero. (2). The total electromotive force in a closed circuit is zero. The first of these laws can be found in Maxwell's formulæ, but it appears there to be the mathematical consequence of an hypothesis concerning the magnetic field produced by an open circuit and not as an experimental fact directly observed. The second law is apparently not included in Maxwell's formulæ. It is interesting to note that these two laws are merely extensions of Kirchhoff's two laws.

The Existence of Rays capable of being Reflected in Radiation Emitted by a Mixture of Chlorides of Radium and Barium.—Th. Tommasina.—Various observations led the author to believe that certain rays existed in the radiation emitted by certain radio-active bodies which were capable of reflection. By using a parabolic concave mirror of silvered copper the rays capable of reflection were separated from the others.

Heat of Formation of Chlorine Hydrate.—M. de Forcrand.—The author employs two methods for finding the heat of formation of chlorine hydrate. (1). A direct measurement of the quantity of heat produced when it is formed, and when it is decomposed; and (2) the calculation of this quantity by means of curves of dissociation. The number deduced from the curves was +18·16 cals.; direct measurement shows it to be +18·57 cals.; the

mean being +18·36 cals. This reaction is capable of being expressed by the equation—

Cl_2 gas + nH_2O liq. = $Cl_2.nH_2O$ sol. . . . +18·36 cals.

The author hopes shortly to establish the exact composition of this hydrate.

NOTES AND QUERIES.

*** Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Extracting Oil from Seeds.—A correspondent asks where he can obtain full particulars regarding the process of extracting oil from seeds by chemical means.

MEETINGS FOR THE WEEK.

MONDAY, 27th.—Society of Arts, 8. (Cantor Lectures). "The Purification and Sterilisation of Water," by Samuel Rideal D.Sc., F.I.C.

TUESDAY, 28th.—Royal Institution, 3. "The Cell—its means of Offence and Defence—Immunity," by Allen Macfadyen, M.D., B.Sc.

—— Society of Arts, 4.30. "To the Victoria Nyanza by the Uganda Railway," by Commander B. Whitehouse, R.N.

WEDNESDAY, 29th.—Society of Arts, 8. "Technical Education as applied to Paper Making," by Clayton Beadle.

THURSDAY, 30th.—Royal Institution, 3. "Recent Excavations at Delphi and in the Greek Islands," by A. S. Murray, LL.D., F.S.A.

FRIDAY, 31st.—Royal Institution, 9. "The Ions of Electrolysis," by Prof. A. Crum Brown, D.Sc., F.R.S., &c.

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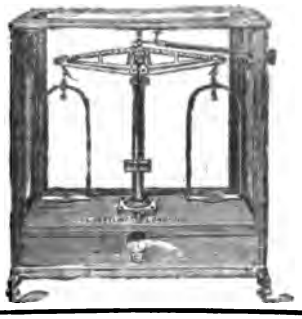

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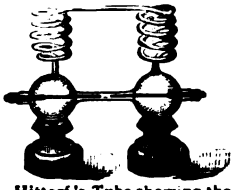

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BRONZING METHODS IN THE ALCHEMISTIC LEYDEN PAPYRI.

By ARTHUR JOHN HOPKINS.

The importance to the history of Alchemy of the Papyri of Leyden V. and X., has been pointed out by M. Berthelot in his "Les Origines de l'Alchimie," and more recently in the "Collection des Anciens Alchimistes Grecs" (1888), in which he presents a translation of the recipes contained in the papyri, and in commenting on them shows their practical nature. In this oldest Alchemistic writing are such recipes for making alloys, and for cleaning the surfaces of the metals, as are used by metal-workers of the present day.

M. Berthelot is the first to show (*a*) that the "dream of alchemy" was, in the begining, not a dream, but a very practical means of deceiving the people by substituting for true gold or silver, crude yellow or silver coloured paints, dyes, and varnishes; coloured alloys of probably recent discovery, and a copper-gold alloy from the surface of which the copper had been removed by "Royal Cement," leaving pure gold in relief; and (*b*) that the alchemists upon deceiving the people fell willing victims to their own deceptions by their ignorance of any individual properties and final tests for the metals, and by their claim of supernatural power in making the precious metals. "It is thus that the artisans, accustomed to make alloys simulating gold and silver, often with such perfection that they deceived themselves, ended by believing in the possibility of really making all sorts of metals by aid of certain combinations of alloys, and certain tricks, completed by the aid of supernatural powers, the sovereign masters of all transformations" ("Collection des Anciens Alchimistes Grecs," I., p. 73).

M. Berthelot is, however, unable to explain all the recipes. Some, which were apparently meant to be the frankest and most in detail, he marks as obscure. Some of course will always remain meaningless, as they are careless shop-recipes, often for secrecy intentionally obscured. But of those which are expressed in detail there still remain a few which seem capable of much meaning if we slightly extend the conception of the ancient worker in metal as presented to us by M. Berthelot. He has pointed out that the Egyptians conceived of silver and gold as a silvery or yellow metallic colour produced by certain synthetic processes. The object of this paper is to add to this conception by establishing the two following theses:—

1st. The Egyptian worker in metal was a bronzer.

2nd. Gold or silver was identified, not only by the colour of the metal, but also by the bronze which could be produced upon it.

The endeavour will be made to show by a detailed study of some of the hundred and one recipes of Papyrus X., together with one from Papyrus V., that these obscure recipes become more intelligible by looking on the Egyptian worker as a bronzer.

This proposition seems almost self-evident on reading the recipes with this changed conception as a guide. With the aid of the second proposition, some recipes become intelligible for the first time.

It is evident that the Egyptians having the bronzer's understanding of the metals would be more liable to self deception, and this would tend further to explain their confidence in the possibility of transmutation.

As M. Berthelot has shown, the existence of "electron" (native alloy of gold and silver), of asem (the artificial alloy of the same metals in varying proportions), of "asem" (factitious, containing no gold or silver), as well as the absence of any exact tests for the metals, compelled the Egyptian artisan to hold the conception of the progressive relativity of the metals rather than the modern conception of strict individuality. Also the religious teaching of the transmigration of souls found additional expression in the transmutation of metals. What then did the Egyptian demand of his metals? Not that they should be modern gold and silver, for our definition of these metals was lacking. Asem, which was considered a metal and played so great a part in the transmutation theory, was sometimes like silver, sometimes like gold,* the property demanded of the metals being one of colour, or metallic sheen. The Egyptian longed for the golden sun-colour with which to adorn his temples, and the metallic colours were both brilliant and permanent. Brilliant colours, and, if possible, permanent colours, the child nations of the world have always delighted in. Gold, silver, purple, and (in Egypt) black are the delights of kings and peoples.

Before beginning the application of these theses to the recipes, it will be well to have a clear statement as to which bronzes are characteristic of silver and which of gold. Under treatment with sulphides in very dilute solution, silver takes a golden bronze, changing gradually to purple, then brown, but finally black. Quick bronzing generally produces an immediate black on silver, which is permanent ("oxidised silver"), and for this blackening of silver Egypt was noted. Pliny states ("Hist. Nat.," xxxiii., 46, Bohn translation):—"The people of Egypt stain their silver vessels that they may see represented in them their god Anubis, and it is the custom with them to paint, not to chase, their silver. This usage has now passed to our own triumphal statues even; and, a truly marvellous fact, the value of silver has been enhanced by deadening its brilliancy." Silver was identified both by its silvery colour and by its black bronze. The production of a black bronze was sufficient to establish the claim for an alloy that it was silver (or nearly silver), or even that it was standard silver ("Chemistry of Moses," 47; Berthelot, "Texte Grec," p. 309). Gold, on the other hand, besides its own colour sacred to the sun, was capable of taking bronzes of most brilliant shades—purple, violet, red, and iridescence.

In the following detailed study of the recipes of the Alchemistic Leyden Papyri from the standpoint outlined in the first portion of this paper, I have chosen the modern work of Arthur H. Hiorns ("Metal Colouring and Bronzing," Macmillan and Co., 1892), as authority for the conduct of different metals under processes as close as possible to those described in the alchemistic recipes. In following these recipes I am depending upon the accuracy of the French translation as presented by M. Berthelot in his brilliant work—the edition of the original Greek not being available. Had this translation been made with the conception of the processes of bronzing as a guide in the mind of the translator, it may be that some of the recipes which have been excluded from the following study could have been used to show still further evidence of the crude beginnings of the bronzer's art.

Papyrus V.—Ἰωσις Χρυσου.

This is the only alchemistic recipe in Papyrus V. It is in two parts as follows:—

"Take some strong vinegar, thicken, take some ———, 8 drachms of common salt, 2 drachms of alum shale, 4 drachms of litharge, rub with the vinegar for three days, separate by decantation, and use. Then add to the vinegar 1 drachm copperas, one-half obole of ———,

* Of the twenty-four recipes in Papyrus X. for the preparation of "asem," nineteen would produce a white alloy, two a reddish-yellow alloy, while three are doubtful.

three oboles of calcite, one and one-half oboles of sory, a silique of common salt, two siliques of salt of Cappadocia. Make a layer with two quarters (of an obole ?). Make it undergo the action of fire . . . until the layer breaks, immediately take the pieces, and regard them as refined gold."

"Having taken four leaves of gold, make foil of them, heat it, and immerse it in copperas rubbed with water, and with other dry copperas, cast (a portion) . . . with the dry material, another with the mixed material, discard the dross, and throw into ——."

The second portion is incomplete. Of the first part, M. Berthelot states that it is cited in Papyrus X., Rx. 15, and that this establishes the connection between the two papyri. As a comparison of these two recipes is necessary, the first important recipe of Papyrus X. is given here:—

Rx. 15.—Colouring Gold.

"To colour gold to make it good for use. Misy and salt and vinegar left from the purification of gold. Mix all, and put into the vase (which contains) the gold described in the preceding preparation. Let it remain some time, and having removed (the gold) from the vase, heat it over charcoal; then put it again into the vase which contains the above-mentioned preparation; do this many times until it becomes good for use."

The title of the recipe in Papyrus V. is given in the original Greek, as there is doubt of its true meaning. M. Berthelot ("Collection des Anciens Alchimistes Grecs," I., p. 13, Note 5) has given four possible meanings for Ἴωσις:—1. Oxidation. 2. Refining. 3. Active principle. 4. Production of a violet colour. In his opinion the word means refining (by oxidation of the base metal of a gold alloy, leaving a surface of pure gold), for he conceives of this recipe as similar to those which define the preparation of the so-called "Royal Cement." He states that the fourth definition, *Production of a violet colour*, though in use with the later (third century) alchemists, is inapplicable here.

This paper will endeavour to show that Ἴωσις means "the production of a violet colour," or bronze (in these papyri as well as later), as indicated by the four following arguments:—

(*a*) Historically it is evident that the original artisan of our papyri was a dyer in purple, for Papyrus X. ends with recipes for dyeing, and the writings of the pseudo-Democritus are prefaced by two careful recipes for dyeing in purple. Also the dyer evidently transferred his dyeing methods to his new business as a worker in metal, for many of the recipes for producing metals of a golden colour strikingly resemble the methods of dyeing. The meaning given last in M. Berthelot's list would, therefore, stand as a link between the early work-shop with its purple dye, and the usage of the later alchemists.*

(*b*) The chemical significance of this recipe called Ἴωσις χρυσοῦ may be seen from a passage in the manual of Hiorns. This author states (p. 152), on the authority of Roberts-Austen, that there is in use in Japan a method for producing upon copper containing some gold (which gold is intentionally introduced into the copper for this purpose), a beautiful rich purple coat or patina. The bronzing or pickling solution contains:—

Verdigris	220 grains
Copper sulphate	540 grains
Water	1 gallon
Vinegar	5 drachms

The presence of a little gold heightens the effect, for, as Hiorns found, copper turns a brownish-red when boiled in this pickle, while copper containing gold becomes purple.

The similarity between this pickle and that of Papyrus V. is significant. We have only to suppose that the workmen were dealing with alchemistic "gold," for which there are many recipes in Papyrus X., rather than with pure gold.

Hiorns states (p. 108) that a solution containing suspended copper hydroxide, and red oxide of iron, produces on copper dipped in it, after removal and heating and numerous repetitions, a violet tint; and that brass (p. 226) dipped in acetic acid solutions tends to give iridescent effects, and that it is possible to arrest the bronzing process at the point of iridescence, and preserve these colours by lacquering (p. 154).

We may conclude that the recipe of Papyrus V. is proper for producing upon cast copper or brass, iridescence; and upon copper containing gold a purple or violet tint; and further conclude that the title of this recipe in Papyrus V. may be translated, *colouring gold violet (with iridescence)*. It would be indeed strange if the Egyptians possessing such a recipe as that of Papyrus V. should have failed to catch the brilliant colours produced upon the copper-gold alloys (Rxs. 56, 87, &c.), which stood for gold in those days, and used it, as M. Berthelot supposes,* for refining only, or for Royal Cement.

(*c*) Pliny's use of the word *virus*. Pliny describes ("Hist. Nat.," xxxiii., 25) a remedy made from gold by melting it with salt and misy and again with salt and schist. The gold remains intact and the other material is the remedy.

M. Berthelot notes that the word *virus* (poison) is the literal translation of the Greek *lós*, which means rust or poison, whence comes the word Ἴωσις, the title of the recipe in Papyrus V. He therefore attempts to show a close relation between this recipe and that of the papyrus, translating this passage "gives active principle to the substances heated with it" (Berthelot, "Collection des Anciens Alchimistes Grecs," I., 15). It is more likely that this passage indicates an attempt to connect all bright coloured substances with medicinal properties, just as the philosopher's stone was considered a universal remedy. The most evident connection between the word *virus* and the Greek *lós* is found in Chapter 23 of the same book of Pliny's "Historia Naturalis," the noted passage concerning electrum, which has been so often cited as evidence of Pliny's credulity. Pliny here states, following the English translation of the Bohn edition: "Native electrum has also the property of detecting poisons (*venera deprehendit*), for in such cases semicircles resembling the rainbow in appearance will form upon the surface of the goblet and emit a crackling noise like that of flame, thus giving a two-fold indication of the presence of poison." With the meaning *poison*, or *active principle* for *virus*, or *lós*, this passage in the "Historia Naturalis" appears absurd. But, as has already been pointed out, *lós* means violet (or purple) bronze, and was so used by the later alchemists. It may be possible that the original Greek word which Pliny translated *stridore* (crackling noise), was *lῳ* dative of *lós* and was mistaken by Pliny for *lωη* (a noise). If this be true, the sense of the passage would be very clear. "Native electrum has also the property of taking a violet bronze, and in such cases semicircular iridescence resembling the rainbow in appearance will form upon the surface of the goblet (in calcibus) together with streaks of violet, like flame."

* If the expression "the preceding preparation" in Rx. 15 is really a citation of the recipe called Ἴωσις χρυσοῦ in Papyrus V., as Berthelot claims, the title of Rx. 15 "colouring gold," and the meaning "colouring violet" would be an additional bond between the two recipes, and therefore between the two papyri. See, however, the argument beyond).

* Hiorns (p. 104) gives a Chinese recipe which he could not make succeed. The pickle consists of copper acetate, cinnabar, ammonium chloride, and alum. If Hiorns had read this in the language of the third century, alum would possibly have been minium, or red oxide of iron, and sal-ammoniac may have been soda, or (as it seems to the writer) more probably borax. (See Berthelot, "Introduction," pp. 30, 45, 237, 251; and Pliny, "Hist. Nat.," xxxi., 39, and xxxi., 46). From experiments in this laboratory, it has been found that this Chinese-alchemistic recipe, so interpreted, gives with sheet copper (if the sheet is heated "until the layer breaks," and then plunged into water) a beautiful reddish-pink colour with wavy greenish lines. Probably the Japanese recipe (*ante*), and the Chinese recipe as just given, owe their origin to Egyptian alchemy.

Here, then, in Pliny is no evidence of credulity, but a clear statement of this wonderful bronze. The passage reminds us strangely of the descriptions given by Hiorns and Roberts-Austen. Pliny, without understanding at all what he was transmitting to us by almost literal translation, here recorded the great discovery of Egyptian alchemists. These workmen, who started as dyers of the royal purple, had begun to produce a purple bronze upon gold. The dyer in purple had become a bronzer in purple. Later, Democritus ("Natural Questions," 4; and "To Leucippus," 5) speaks of this bronze as "crysocorail," shell of gold, just as bronzers now speak of the patina, and *lós* means violet, as Zosimos dreams of the priest whom he named *Ἴον* in the temple ("Zosimos," Lesson I., 2).

(*d*) In the acme of European alchemy, in the thirteenth century, when, on account of the possibility of closely identifying the metals, colours were no longer considered exponents of the precious metals or the great desideratum, we find the pseudo-Albertus, the charlatan, ending his description of processes inherited by him from the Egyptian workshops with promises of the victory coming to the alchemist as the reward of patient toil; "the mass is changed into a very red stone, which philosophers call blood or purple, red coral, red sulphur." This thirteenth century praise of red and purple, the "Philosopher's Stone," unintelligible to "Albertus," was but an inheritance from the Egyptian dye and bronzing works where purple, the royal colour, was produced from gold, the royal metal; and purple was the tincture* of gold; and metals which could produce a bronze of purple, violet, iridescence or even red were considered gold or nearly gold.†

The thirteenth century alchemists carry their mysterious processes through the black and white and yellow of the early alchemists (Olympiodor, "Sacred Art," 37-40, and Berthelot, "Les Origines de l'Alchimie." p. 242), but in the *finale* give purple and violet as the highest attainment of the art; in the Japanese and Chinese bronzing works, these processes for the production of purple have been preserved, untouched by European alchemy and just as they came from Egypt, for the use of our bronzers of to-day.

With the meaning of *Ἴωσις* established, the recipe in Papyrus V. becomes a recipe for bronzing, and we can turn to Rx 15, to see what light our notions of bronzing will throw upon it. It is not very clear why the two expressions, "la préparation précédente" and "la préparation susdite" should refer back past Papyrus W. to Papyrus V., as M. Berthelot suggests. The "preceding preparation" is certainly given in Rx 14, entitled "Making the mixture for a preparation," which gives directions for preparing a white alloy, like "bell metal," of copper and tin. This is "Asem" (as in Rx. 8), which M. Berthelot has shown by abundant illustrations was confused with gold and called *χρυσος λευκός* (Berthelot, "Collection des Anciens Alchimistes Grecs," I., p. 31, Note 1). It would seem very reasonable that this white "asem" should be "the mixture (alloy) for a preparation" *of gold* by adding it to gold or to real asem or to copper, as in Rxs. 6, 8, and 56, this being common alchemistic practice. But if the "gold" of Rx. 15 is the copper-tin alloy of the preceding paragraph, the pickle described in Rx. 15 would produce upon it a fine yellow bronze (Hiorns, p. 107). If it is to be understood that this alloy was first to be added to some gold, the bronze would probably be purple with iridescence, for a second Japanese recipe quoted from Roberts-Austen (Hiorns, p. 152), to be used upon copper containing some gold, contains exactly the ingredients of Rx. 15 with the addition of sulphur and nitre. M. Berthelot's supposition that this Rx. 15 and the one in Papyrus V. are similar to the recipes for Royal cement is inadequate to the case. Rx. 25 is much more exactly the Royal cement, as it contains no acid, and the process is conducted in closed vessels.

But Rx. 25 is entitled "Gold Varnish," which Royal cement really is, as Macquer describes it (Berthelot, "Introduction," p. 15). As this title is correct, so probably is that of Rx. 15 "Colouring Gold"—the colour being that for which gold was noted, purple with iridescence. But this supposition of M. Berthelot's would further involve the separation of the two Rxs. 14 and 15, which is very improbable. The expression in Rx. 15, "vinegar left from the preparation of gold," would seem to indicate the "preparation" (of gold) "above mentioned" in the title of Rx. 14, and that this recipe was a continuation of that process, a continuation in this sense:—Rx. 14 describes the making of "Asem," which was confounded with gold not because of its colour, for this preparation is white (see note, *ante*), but because it acted like gold (especially when a little [ferment of] gold was added to it) in assuming permanent and brilliant bronzes, such as are formed on fine gold; then Rx. 15 shows how to produce these colours—yellow, violet, purple, or iridescence. The colour was the important thing, In just this alchemistic sense of the importance of colour, Mary the Jewess says (Olympiodor, "Sacred Art," 40): "Our lead is artificially black . . . common lead is fundamentally black." The property of blackness was a more essential thing than lead or silver, just as liquidity was considered an entity present most largely in lead and mercury. So Rx. 14 directs the formation of that alloy which could produce and hold the essential (*i.e.*, colour) of gold, if treated as immediately directed in Rx. 15.

Rx. 16.—Increasing Gold.

"To increase gold, take Thracian cadmium; make the mixture with caked cadmium or that of Gaul."

Rx. 17.—Imitation of Gold.

"Misy and sinope red, equal parts to one part of gold. After having put the gold into the furnace and it has become of a fine colour, cast upon it these two ingredients, and removing (the gold) allow it to cool, and the gold is doubled."

M. Berthelot thinks Rx. 17 is a continuation of Rx. 16, because he considers Rx. 16 incomplete, and that the separating title, "Imitation of Gold," is a later marginal commentary which has crept into the text. It is probable that Rxs. 16 and 17 bear the same relation to each other as was pointed out for the couplet Rxs. 14 and 15. The first recipe in each is complete in itself in that the mixture or increase has been accomplished, but the product "asem" is only potentially gold and the colouring follows in each case, because that was the original intention in forming the alloy in the first process. In the absence of textual evidence, the title, "Imitation of Gold," would not then be considered a gloss. The titles of the recipes seem to be singularly correct and suggestive (see *ante*). The original Greek was not numbered, and a modern compiler would probably have made a separated couplet of the two Rxs. 14 and 15, and another of Rxs. 16 and 17 to bring out the clear thought. Rx. 16 would give us a light-coloured alloy of gold, zinc, copper, and lead. Treated as directed in Rx. 17, with sulphates of iron and copper, and either red oxide of iron or minium at a temperature at which the bright colour of gold and brass had begun to appear, copper would bronze a brown or red colour (depending on temperature and time), as we know from the results of similar recipes

* See Berthelot, "Collection des Anciens Alchimistes Grecs," I., p. 267, under the words "Trempe-teinture," for the different meanings of this word in use among the alchemists.

† M. Berthelot states that though the later alchemists worked with coloured glasses, no recipes for this kind of work are found in Papyrus X. This papyrus was probably written upon the discovery of brilliant and permanent metallic colours similar to purple dye. The discovery of the method for making red and purple glasses probably antedated this papyrus, and by the time of the Democritan writings, recipes for coloured glasses became important to the alchemists. One of the first recipes for making a red glass is described in alchemistic language in "Technical Treatises," v.,6 (Berthelot, "Traduction," p. 333-4), in which after making green, yellow, white, and blue glasses, by mixing and rubbing and other processes, as the climax a red "cinnabar" or "gold" is produced.

given by Hiorns (Pp. 98-101). But the presence of the other metals would vary the effect, which would tend, as far as the influence of the gold was concerned, towards purple, as has been already pointed out.

The position taken in reference to these two recipes (16 and 17), is confirmed by Rxs. 87 and 88, which are practical copies of Rxs. 16 and 17. The first (Rx. 87) is called "Doubling Gold," and begins "To increase the weight of the gold." The second (Rx. 88) begins "One alters gold, on increasing it, with misy . . . &c." and ends "The gold is doubled." Then Rx. 89 follows with another method of bronzing, quite different from the method of Rx. 88.*

Rx. 36.—*Making Asem Black like Obsidian.*

"Asem two parts, lead four parts. Put in an empty earthen vase. Throw on it a triple weight of raw sulphur, and having put it in the furnace, melt. And having taken from the furnace, make a casting and form what you wish. If you wish to make an image in metal stamped or moulded, then polish and engrave. It does not rust."

Rx. 49.—*Gilding Silver.*

"In order to gild, without (gold) leaves, a silver or copper vase, dissolve some yellow soda and some salt in water. Rub with this, and it will be gilded."

Rx. 89.—*Another (process).*

"Invention of sulphur water (or holy water). A handful of lime and as much sulphur in fine powder. Put them in a vase containing strong vinegar or the urine of a young child. Heat from below until the supernatant liquid appears like blood. Decant the latter carefully to separate it from deposit and use."

Rx. 36 shows the importance attached to black. Black was the origin of all colours. Liquidity was the origin of all solids. Lead which was black and easily liquefied was at first the elementary metal until mercury supplanted it. Silver from its black bronze was allied to lead. (Olympiodor, "Sacred Art," 38). Asem was like silver or like gold either by its original white or yellow colour, or by its bronze, black, or purple. In the case of Rx. 36 we have a substance bronzed black, not on the surface only, but black through and through. It would not rust. It had a permanent colour. It was better than "oxidised silver." Rxs. 49 and 89 involve the use of soluble sulphides. M. Berthelot has shown that "yellow soda" is probably sulphide of soda, but characterises the recipe as obscure. The following quotation from Hiorns (p. 266), will remove this obscurity.

"The following is a most valuable solution for colouring silver goods:—

Barium sulphide	5 grains
Water	5 fluid ounces

This solution imparts a beautiful golden tint to silver, and when worked cold, almost any desired shade from pale to deep gold may be obtained according to the time of immersion. If the immersion is continued, the gold colour passes through crimson-purple to brown, the former being more or less iridescent. . . . To produce the gold colours, it is best to work with a cold solution, because the changes succeed each other more slowly, and the process can be stopped when the desired shade of colour is obtained."

Here we have white silver passing through the yellow colour of gold and on to crimson-purple with iridescence. First silver, then gold, last the "coral of gold." This was the highest of colours—the *lôs*.

Rx. 89 gives the method for making "oxidised silver" by the use of a solution of soluble sulphide, stronger than the very dilute solution of Rx. 49. This recipe follows Rx. 88, which has already been shown to describe a bronzing process.

Rx. 64.—*Test for Asem.*

"To tell whether asem is counterfeit. Place in brine, heat; if it is counterfeit, it blackens."

In the case of this recipe the apparent obscurity is removed by a reference to Hiorns. He directs (p. 124). "Dissolve the copper chloride in the water, raise to the boiling-point, and immerse the previously well-cleaned copper articles for a few minutes. If the copper is pure or nearly pure, a very beautiful rich reddish-brown colour will be obtained, but with ordinary commercial copper containing 1 to 2 per cent of impurities the colour obtained is nearly black."

This was, therefore, a test for the foreign metals (other than tin) associated with copper, depending upon the production of the clear brown bronze characteristic of copper.

M. Berthelot has marked this recipe obscure, with nearly all the other recipes which have any alliance with bronzing methods. But by regarding the Egyptian artisan of these papyri as a bronzer, by accepting the fact that gold and silver were identified not only by the colour of the metals but also by the colour of the bronze they were capable of taking, the recipe given becomes at once clear.

In final justification of the two theses this paper has endeavoured to assert, many of the remaining recipes of Papyrus X., read from this changed point of view, become plainer and represent intelligent and workmanlike directions for producing either alloys capable of bright bronzes or bronzing solutions into which these alloys were to be dipped.

Amherst College, Massachusetts.

* Perhaps Rxs. 87, 88, and 89 should be in a separate group and the titles of Rxs. 88 and 89 translated by Berthelot "*Autre*," and "*Autre*" would indicate what we would express by such an expression as: After Rx. 87, choose either of the two following elective processes. (See beyond for Rx. 89).

RESEARCHES ON THE PRESSURE FORCES OF LIGHT.*

By PETER LEBEDEW.

(Continued from p. 40).

III. *The Experiments.*

The methods described above allow two fundamental questions to be answered experimentally:—

i. Whether light radiation can exert forces which are independent of the known secondary (convective and radiometer) forces, and

ii. Whether these "weight moving" forces of light correspond to the pressure forces calculated by Maxwell and Bartoli.

Before the beginning of the experiment an auxiliary thermo-element (which was connected with a d'Arsonval's galvanometer) was moved along the direction of the axes of both lenses L_1 and L_2 (Fig. 1), and their focal lengths for the principal ray observed. Then the mirrors and the lenses of the apparatus (Fig. 1) were so adjusted that the real images of the diaphragm formed by the rays coming from the right and from the left fell at the same place.

In order to compare the intensity of both beams of light the thermo-element was placed where the real images were formed, and alternately illuminated from right and left. Usually only a small difference (scarcely reaching 1 per cent) was found in a great number of experiments; in the case of most reflecting glass surfaces an unequal distribution of dust is sufficient to account for such a difference.

If the the thermo-element is moved $\pm 0{\cdot}5$ c.m. from its original position in the direction of the axis of the cone of light, the light-intensity diminishes for both directions

* From the *Annalen der Physik*, 1901, vol. vi., p. 433.

Table I.

Apparatus III. Platinised Vane (2). Distance of Centre of Disc from Axis of Rotation $a = 9{\cdot}2$ m.m.
Cooled with Ice and Salt. Distance of Scale A = 1195 div.

R.		Calc.		L.		Calc.		R.		Calc.		L.		Calc.		R.
		306		115						307		174				—
176	240				206	295		184	245				210	244		—
	239	302		118	207				244	303		177	211			—
177	239				208	296		184	243				212	245		—
	240	302		124	209				243	300		180	213			—
178						294		189							247	—
	240				208				244				212			
Deviation			32 div.				36 div.				32 div.					—
	Gb.				Gn.				Gb.				Gn.			Gb.
	308								314							—
	305								312							—
	312				201				314				201			—
	314								316							—
	310								314							
Galv.			109 div.				113 div.				113 div.					
Red. def. (G = 100)..			29·3 div.				31·8 div.				28·3 div.					

In this Table—
R and L denote the turning points on the scale if the light falls from right (R) or left (L).
"Calc." in the middle column gives the position of rest calculated from three adjacent turning points.
"Deflection" gives the deflections of the system if the light is changed from right to left.
Gb and Gn give the positions of the galvanometer if the thermopile is illuminated or not respectively (galvanometer zero-point).
Galv. gives the galvanometer deflections.
Red. def. (G = 100) are the above named deflections reduced to a constant galvanometer deflection of 100 div.

of the rays by about 5 per cent. The preliminary investigations described are absolutely necessary.

The vane apparatus was so suspended that the rays coming from the lamp, which were reflected from the concave glass wall of the globe, could not illuminate parts of the vane apparatus.

After the vane apparatus was suspended the globe was closed, and for several days highly exhausted, heat being gently applied at the last to the whole glass covering, the single vanes being illuminated. Before each series of experiments the lower part of the globe containing the mercury was warmed in the water-bath to about 5° C. above the temperature of the room,* exhausted for one to two hours, then closed by the mercury in v (Fig. 5), and cooled by a mixture of ice and salt.

The chief error of the measurements is caused by disturbances originating in convection; these disturbances show themselves in constant movements of the zero-point, the magnitude and direction of which depend on accidental circumstances;† however, they are fairly small during the taking of one measurement, and may be eliminated by a long series of observations; convection of the residuum of mercury vapour is brought about both by the warming of the illuminated vane and by unequal warming of the walls of the globe, and especially by temperature differences of the two mercury surfaces. If an attempt is made to take observations without cooling the mercury considerable‡ disturbances appear owing to convection; these become much smaller when a freezing mixture is applied.

Another source of error lies in the inconstancy of the source of light;§ sudden fluctuations of the light-intensity cause the amplitude of oscillation of the vane apparatus to alter by leaps and bounds; they can only be avoided by taking many observations.

The observer could alternately read off the vane apparatus and the galvanometer through two conveniently placed telescopes; an assistant who attended to the arc lamp could shift the mirrors s_1 s_4 (Fig. 4) at bidding. By intermittent illumination the amplitude of oscillation of the vane apparatus could be brought to any desired magnitude.

Table I. gives the commencement of a record of observations.

As shown in Table I. seven R and L observations were taken for each vane, and the "def. red." obtained were united to a mean specifying the mean $\pm$ divergence of all observations from that mean. (For the vane of Table I. the double deviation = $29{\cdot}4 \pm 1{\cdot}6$ div.).

In order to be able to compare the measurements, which were made with individual discs, more easily at sight, certain calculations and corrections are necessary.

In the apparatus I. and III. a band of light (breadth about 3 m.m.) falls on the supporting arms, and this increases the deflection. By measuring the illuminated parts and their axis distance this amount, which in different vanes varies from 5 to 10 per cent, may be calculated and deducted; thus we get the deflection brought about by the circular discs alone. (Apparatus II. does not need this correction). For the vane of Table I. this correction amounts to 1·9 div.; the calculated double deflection would be 27·5 div.

The measurements of the distances of the mid-points of the discs from the axis of rotation were accomplished as follows:—The mirror apparatus (Fig. 1) was drawn back on the sliding base, a plumb line of thin silver wire was hung close in front of the globe on the side of the incident light, a telescope was placed perpendicular to the vane arms about 4 m.m. away, and the wire moved until it appeared to coincide with the torsion thread. The telescope had a micrometer eye-piece, which was read by the help of a scale placed close to the globe; the apparent distance of the disc centre from the silver wire, which

* With this small temperature difference no mercury is deposited on the colder parts of the apparatus, a phenomenon which was pointed out by M. Cantor in the case of surfaces not capable of being moistened.

† In researches with the same vane on different days the magnitude and direction of the deviations of the zero point could appear very different.

‡ At higher pressures the observations are rendered very uncertain by convection, and scarcely admit of reliable measurements.

§ Siemens's "A Carbons" have proved very good; with inferior carbons it is scarcely possible to take measurements.

corresponds to the true distance of the axis of rotation, could be exactly observed to 0·5 m.m. The magnitude of these distances lay between 9 and 11 m.m.

On the basis of these measurements the observed deflections were reduced to a deflection corresponding to an axis distance of 1 c.m. For the vane of Table I. the reduced double deflection amounts to 29·9 div.

In order to express in absolute measure the magnitude of the observed pressure which the light exercises on the vane under examination, a measurement of the direction force of the suspension thread was taken by means of observations of the oscillations, while the periods of oscillation were obtained from three series of observations of ten oscillations.

TABLE II.

Half oscillation period.	Inertia body. Copper-wire.
Mirror alone .. $\frac{t_1}{2}$ = 5·1 ±0·05 sec.	Length = 4 c.m.
Mirror + inertia body .. .. $\frac{t_2}{2}$ = 29·4 ±0·1 sec.	Mass = 0·314 grm.

Direction force D = 0·00494 dynes.

Based on this direction force we obtain for the vane of Table I. the magnitude of the pressure for partial illumination expressed in dynes—

p = 0·0000308 dynes ± 0·0000017 dynes.

In order to prove the relation stated by Maxwell and Bartoli we must *calculate* the pressure forces which the light radiation of the experiments must exert according to Maxwell and Bartoli, and this calculation must then be compared with the result of the experiments. For this a calorimetric measure of energy and a photometric determination of the reflecting power are necessary.

(To be continued).

EXPERIMENTS RELATIVE TO THE CONSTITUTION OF PECTOLITE, PYROPHYLLITE, CALAMINE, AND ANALCITE.*

By F. W. CLARKE and GEORGE STEIGER.

(Continued from p. 47).

Pyrophyllite.

THE empirical formula for pyrophyllite, $AlHSi_2O_6$, is apparently that of an acid metasilicate, and the mineral is therefore peculiarly available for fractional analysis. The compact variety from Deep River, North Carolina, was taken for examination, and a uniform sample was prepared. Analysis gave the following results :—

SiO_2	64·73
TiO_2	0·73
Al_2O_3	29·16
Fe_2O_3	0·49
MgO	trace
Ignition..	5·35
	100·46

If, now, pyrophyllite is an acid metasilicate it should break up on ignition in accordance with the equation—

$$2AlHSi_2O_6 = Al_2Si_3O_9 + SiO_2 + H_2O;$$

that is, one-fourth of the silica, or 16·18 per cent, should be liberated. The mineral itself is very slightly attacked by boiling with the sodium carbonate solution, and in an experiment of this kind only 0·72 per cent of silica was dissolved. Upon ignition under varying circumstances the following data were obtained :—

Ignited ten minutes over a Bunsen burner, and then extracted with sodium carbonate solution, 1·51 per cent of SiO_2 dissolved.

Ignited fifteen minutes over a Bunsen burner, 1·89 per cent became soluble.

Ignited ten minutes over a Bunsen burner and then fifteen minutes over the blast, 2·84 per cent of silica was liberated.

These results are of a different order from those given by pectolite and talc, and raise the question whether pyrophyllite, despite its ratios, is a metasilicate at all. So far as the evidence goes, it may with propriety be regarded as a basic salt of the acid $H_2Si_2O_5$, and its formula then becomes—

$$Si_2O_5 = Al—OH.$$

This formula is at least as probable as the metasilicate expression, which latter rests upon assumption alone. Still other formulæ, but of greater complexity, are possible; but until we know more of the genesis and chemical relationships of pyrophyllite, speculation concerning them would be unprofitable.

By heating with dry ammonium chloride, pyrophyllite is very slightly attacked. In two experiments it lost in weight 6·17 and 6·30 per cent respectively. The excess of loss over water is due, as we have proved, to the volatilisation of a little ferric and aluminic chloride. The residue of the mineral after this treatment contained no chlorine, so that no chlorhydrin-like body had been formed. The formation of such a compound, the replacement of hydroxyl by chlorine, would, if it could be effected, be a valuable datum toward determining the actual constitution of the species.

Calamine.

The simplest constitutional formula for calamine, the one which is generally accepted, represents it as a basic metasilicate, $SiO_3 = (ZnOH)_2$. In this the hydrogen is all combined in one way, and so, too, is the zinc. In all other possible formulæ, simple or complex, the hydrogen as well as the zinc must be represented as present in at least two modes of combination; a condition of which, if it exists, some evidence should be attainable. Our experiments upon calamine have had this point in view; and we have sought to ascertain whether water or zinc could be split off in separately recognisable fractions. Our results, in the main, have been negative, and tend toward the support of the usual formula; but the data are not conclusive, although they seem to be worthy of record.

The beautiful white calamine from Franklin furnace, New Jersey, was selected for study, and gave the subjoined composition :—

Analysis.

SiO_2	24·15
$Al_2O_3.Fe_2O_3$	0·19
ZnO	67·55
CaO	0·12
H_2O	7·95
	99·96

Fractional Water.

At 100°	0·27
At 180°	0·22
At 250°	0·75
At 300°	0·88
Incipient red heat	4·46
Full red heat	1·37
	7·95

Here no clear and definite fractionation of the water is recognisable, at least of such a character as to suggest any other than the ordinary formula for calamine.

Upon boiling powdered calamine with water practically nothing went into solution, but by boiling with the solution of sodium carbonate 0·25 per cent of silica was dissolved. After ignition at a red heat, only 0·14 per cent of

* *Bulletin of the United States Geological Survey*, No. 167.

silica became soluble in sodium carbonate, and after blasting, only 0·24. In these experiments a very little zinc was dissolved also; but there was no evidence that any breaking up of the mineral into distinguishable fractions had occurred. In a hot 10 per cent solution of caustic soda both the fresh and the ignited calamine dissolve almost completely; but boiling with aqueous ammonia seems to leave the mineral practically unattacked. All experiments aiming to extract a definite fraction of zinc while leaving a similar fraction behind resulted negatively.

By heating with dry ammonium chloride, calamine is vigorously attacked, and gains in weight by absorption of chlorine. In two experiments the mineral was intimately mixed with three times its weight of powdered sal-ammoniac and heated in an air-bath for several hours to a temperature somewhat over 400°. A large part of the residue was soluble in water, and the percentage of this portion, together with the percentage increase in weight, ig given below.

	I.	II.
Gain in weight	27·60	25·78
Soluble in water..	53·23	67·13

A conversion of calamine into the chlorhydrin, $SiO_3(ZnCl)_2$, would involve a gain in weight of 15·34 per cent. Complete conversion into $2ZnCl_2+SiO_2$ implies an increase of 38·14 per cent. The figures given lie between these two; and are indefinite also for the reason that there was volatilisation of zinc chloride.

In two more experiments the calamine, mingled with three times and four times its weight of ammonium chloride respectively, was heated for an hour and a half to bright redness in a combustion tube. The zinc chloride which was formed volatilised, and was collected by suitable means for determination. It corresponded to 59·6 and 59·0 per cent of the original mineral, calculated as zinc oxide; which indicates a nearly complete decomposition of the calamine into $2ZnCl_2+SiO_2$. The residue was mainly silica, with a small part of the zinc, about half of the silica being soluble in sodium carbonate solution. Here, again, no definite fractionation of the mineral could be observed.

Finally, the action of dry hydrogen sulphide upon calamine was investigated. The mineral was heated to redness in a current of the gas, and gained perceptibly in weight. The percentage data, reckoned on the original calamine, were as follows, in two experiments:—

	I.	II.
Gain in weight	6·00	6·43
SiO_2 soluble in Na_2CO_3 ..	16·45	20·95
Sulphur in residue	—	24·12

Complete conversion of calamine into $2ZnS+SiO_2$ implies a gain in weight of 5·80 per cent; and it is therefore evident from the figures of the second experiment that the limit of change was approached very nearly. The 24·12 of sulphur taken up is quite close to the 26·53 per cent which is required by theory. About eight-ninths of the calamine had undergone complete transformation. Again, no definite fractionation was detected.

The hydrogen sulphide reaction was examined still further with reference to the temperature at which it becomes effective. Even in the cold calamine is slightly attacked by the gas, but its action is unimportant until the temperature of 400° is approximated. Then it becomes vigorous and the reaction goes on rapidly. A few experiments with willemite showed that it also was attacked by hydrogen sulphide, but less vigorously than calamine.

(To be continued).

The Sanitary Institute.—A donation of one hundred guineas has been made by the Vinolia Company to the Fund for the new Building and Endowment of the Parkes Museum.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, January 16th, 1902.

Prof. EMERSON REYNOLDS, Sc.D., V.P.R.S., President, in the Chair.

MESSRS. Jollyman, Kettle, and J. H. Allworthy were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Edwin Bayles Atkinson, Scartho House, Great Grimsby; Matthew Bradbury Challen, Daylesford, Victoria; Noël Heaton, 20, Girdlers Road, West Kensington, W.; William Brannan Jackson, Glengowan, Caldercruix, N.B.; Selwyn Philip James Levelle, Royston Villa, Ashby Road, Burton-on-Trent; Allan Ogilvie, 19, Market Street, Millom, Cumberland; Nevil Vincent Sidgwick, Lincoln College, Oxford; James Swain, 17, Winsham Street, West Side, Clapham Common, S.W.

Day and Hour of Meeting.

The Council has decided, on the recommendation of the Committee appointed for the purpose, that for the remainder of the Session the Ordinary Meetings of the Society shall be held at the following times:—

January: Thursday, Jan. 16, at 8 p.m.
February: Thursday, Feb. 6, at 8 p.m.; Wednesday, Feb. 19, at 5.30 p.m.
March: Thursday, March 6, at 8 p.m.; Wednesday, March 19, at 5 30 p.m.
April: Thursday, April 17, at 8 p.m.; Wednesday, April 30, at 5.30 p.m.
May: Thursday, May 15, at 8 p.m.; Wednesday, May 28, at 5 30 p.m.
June: Thursday, June 5, at 8 p m.; Wednesday, June 18, at 5.30 p.m.
Annual General Meeting, Wednesday, March 26th, at 4.30 p.m.

Of the following papers, those marked * were read:—

*1. "*An Investigation of the Radioactive Emanation produced by Thorium Compounds.*" I. By E. RUTHERFORD, M.A., D.Sc., and F. SODDY, B.A.

The main questions examined by the authors are:—(1) Can thoria, the emanating power of which has been largely destroyed by ignition, have this property restored to it by chemical treatment? (2) Is emanating power to be regarded as a specific property of thorium, or is it due to the presence of a foreign material? (3) Does the radioactive emanation itself resemble in chemical nature any known substance?

The electrical methods employed for the measurement of emanating power and radioactivity depend on the universal property of the radiations from substances known as radioactive of ionising or producing charged carriers from the gas through which they pass whereby the gas is rendered capable of conducting a current of electricity. The amount of the current, measured by means of a quadrant electrometer, affords the means of comparing the intensities of radiations, and the method is capable of a degree of precision and rapidity impossible with the better known photographic method. Use was made of the rate of decay of the radiation from the emanation with lapse of time to recognise and distinguish between different types of emanation, and the study of the phenomena is thus rendered qualitative as well as quantitative in character. The means employed for eliminating the effects of the emanation from the effects of the straight line radiation and *vice versâ* were explained.

The first question is conclusively answered in the affirmative. After solution and re-precipitation, no difference was observed in the emanating powers of thorium compounds prepared from ordinary thoria, and thoria de-emanated as

completely as possible by intense ignition. The latter process must therefore be regarded as an obliteration of the effect rather than a removal of the cause. The de-emanation of thoria by ignition leaves the straight line radioactivity quite unaffected.

With regard to the second question, the attempts first made to separate from thorium a substance to which the emanating power could be traced were not successful. These included a fractionation of the sulphate and a precipitation of thoria by potassium azoimide according to the method of Dennis. No change in either the emanating power or radioactivity was observed in thoria prepared by these methods. Subsequently, results of a different character were obtained.

It was found that water vapour increases, and desiccation decreases the normal value of the emanating power, the extremes varying in the ratio of 2 to 1. Cooling acts powerfully in the opposite direction to increase of temperature. At −78°, the emanating power was only one-tenth of the normal, but no permanent alteration in value is produced. The lapse of time in increasing the emanating power of freshly prepared thorium hydroxide is very striking. A maximum value appears to be attained after some days, and this is not much affected by sealing up the substance in glass.

An investigation of the third point, the chemical nature of the emanation itself, showed that this possesses the property of chemical inertness which characterises the members of the argon family. Using in the different cases suitable gases to carry the emanation from the thoria, it was subjected to the action of the following reagents on its way to the testing apparatus:—Red-hot lead chromate, white-hot platinum, platinum-black raised gradually to a white heat, red-hot magnesium, zinc dust, and palladium black. In each case the radioactivity of the gas current was quite unaffected, proving that, not only is the emanation not absorbed by these reagents, but also that its radioactivity is not thereby affected. The possible explanation that the emanation is the manifestation of excited radioactivity on the surrounding atmosphere was shown to be untenable by a crucial experiment. Since the only known gases which would survive in unaltered amount the action of all the reagents employed are the gases of the argon family, the authors conclude that the emanation is allied in properties to the elements of this group.

The emanating power of thorium nitrate, which in the solid state is only 1 8 per cent of that of thoria, is increased 200 times by solution in water. It was also found that the emanating power of the carbonate varies enormously with the method of preparation, and specimens of it have been obtained differing in the ratio of 80 to 1, of which the highest possesses four times the value of thoria.

A remarkable observation has been made on the solutions from which thorium has been precipitated with ammonia; although these should be free from thoria, they still possess both emanating power and radioactivity in considerable amounts. This has led to the preparation of substances free from thorium, but possessing radioactivity and emanating power indistinguishable from that of thorium, many hundred, in some cases over a thousand, times as active. The identity extended to the penetration power of the rays for metals and the rate of decay of the emanation. The fact that those processes in which a portion of the emanating material is certainly removed from thorium does not much affect the value of the emanating power of the latter, taken in conjunction with the very great variations which this value suffers in different circumstances and often spontaneously with time, leads to the belief that emanating power is the manifestation of a dynamical change rather than a function of matter in the static condition. In the same way as in chemical reaction, the active mass of a reacting solid appears independent of its quantity, so the rate of the change, of which emanating power is a measure, does not depend solely or even mainly on the quantity of emanating material present. The removal of the latter is consequently not accompanied by corresponding changes in the emanating power. The straight line radioactivity, on the other hand, is apparently unaffected by any of the causes which so profoundly alter the value of the emanating power, and appears much more likely to be additive with regard to mass. The effect of the removal of an intensely radioactive and emanating material from thorium is found, in accordance with this view, to reduce the straight line radioactivity of the thorium very materially. These results show that thorium is partially separated from ThX, the constituent responsible for its activity, and the latter can be obtained free from thorium.

The chemical examination of the thorium-free active substances prepared in the manner described showed the presence of an unknown substance, precipitated from its solutions by sodium phosphate. This the authors regard as merely an accidental impurity. The actual ThX is probably present only in minute quantity, exhibiting the behaviour of being dragged down by precipitates, without reference to its true analytical behaviour.

Another method of separation confirms this view. By evaporation of water with which thoria has been shaken for some time, small deposits are obtained which are often of the order of a thousand-fold more active than the original thoria. The washed sample exhibits a considerable reduction of radioactivity after the process, whilst the character of the radiations as before is identical with the ordinary thorium radiation. A careful chemical examination failed to reveal the presence of any other element besides thorium. The view that the radioactivity and emanating power of thorium is to be ascribed to the presence of a minute amount of a constituent ThX of correspondingly great activity is thus confirmed.

Discussion.

Mr. Vernon Harcourt had previously read two papers by Professor Rutherford in the *Philosophical Magazine* which explained the apparatus used by the authors. The authors' conclusion that the influence upon the conducting power of air or other gas interposed between oppositely electrified surfaces is due, not to an imponderable force radiating from the molecules of thoria, but to the escape in minute quantity of some substance which is mixed with the thoria, and which can be dissolved and precipitated, was of very great interest. He might recall the observations and conclusions of Dr. Russell, who had investigated the photographic effect of the emanation from a number of substances, and had similarly concluded that the results were due, not to such an agent as light or electricity, but to the action of hydrogen peroxide.

(To be continued).

PHYSICAL SOCIETY.

Ordinary Meeting, January 24th, 1902.

Prof. S. P. Thompson, President, in the Chair.

A paper on "*The Factors of Heat*" was read by Mr. James Swinburne.

In all branches of physics, except heat, energy is divided into pairs of factors. Heat is generally thought of as a sort of indivisible energy and is not split into factors, but is treated as a whole, so that we have conductivity for heat, capacity for heat, specific heat, &c. Capacity for heat and specific heat are also taken when they include external work, at constant pressure for instance; so that the capacity is reckoned as capacity for energy which is only partly in the body or substance. So little is heat realised as energy that it has its own unit, so that equations involving other forms of energy with it need to be complicated with a coefficient. Temperature might be a factor of heat; but there is no corresponding quantity

factor. There is no unit of temperature; it is measured in degrees which have no proper connection with anything. Temperature is sometimes treated as a tension factor with heat as the quantity factor, as when heat is said to run down temperature. Heat is thus regarded as its own quantity factor. Entropy is sometimes incorrectly used as the quantity factor corresponding to temperature. Entropy is at present indispensable as a function involving information as to whether heat has been, or might have been, converted into work. The author discusses "chy" as a possible factor for use with absolute temperature where "chy" is a quantity factor, such that when multiplied by the temperature at which it is added or withdrawn gives the energy added or withdrawn. In the θ, χ system capacity, specific capacity and conductivity vary inversely as the temperature. These factors are not analogous with the factors of other forms of energy, and are not convenient. The energy of heat is therefore split into $\tau\ \pi$, where τ is proportional to the square root of the temperature, and is called by the author "tasis." The other factor, π, is called "posot." In any gas, tasis is proportional to the effective velocity and posot to the momentum. Tasis and posot are analogous to the tension and quantity factors already in successful use and indispensable in the treatment of other forms of energy. Conductivity of posot follows Ohm's law, and the capacity of a body for posot is constant.

Prof. Everett said the author of the paper had made a bold attempt to put the theory of heat on a better foundation. One objection to his proposals was that the two new variables, tasis and posot, were not independent, at all events in dealing with a perfect gas. The energy of the gas being $\frac{1}{2} mv^2$, tasis was represented by v and posot by mv. There was thus only one independent variable (for m is a constant mass), and arbitrary variations in the condition of the gas could not be represented. Another objection was the dubious nature of posot. Posot was described as momentum; but the total momentum of the gas was zero; its centre of gravity being at rest. Momentum as a determining element in physical phenomena was always a vector, and yet posot was a scalar. It was not even the mass mulpiplied by the arithmetical average of velocity; for v was not the average of velocities, but the velocity of mean square. The claimed simplification of the law of conduction rested on the erroneous assumption that the conductivity of a metal is inversely as its absolute temperature. He thought the proposed changes involved loss of simplicity without compensating advantage, and had been suggested by an undue desire to imitate electrical methods in the treatment of heat.

The Secretary read a letter from Prof. G. H. Bryan, in which he said that the author's system was very difficult to understand from his paper, but when reduced to its simplest terms it appeared to possess great disadvantages in comparison with the conventional method of thermodynamics. Mr. Swinburne's proposal to put $dU = Tdx$, where x is what he calls "chy," had the disadvantage that dx was not a perfect differential, and the value of x for any body depended not only on its actual state but also on its past history. The same thing occurred when tasis and posot were used, as $d\pi$ was not a perfect differential, and the two quantities seemed more inconvenient than chy. The paper overlooked points of vital importance, and contained a great deal of irrelevant matter about stand-pipes and reservoirs. It would be interesting to know whether the heat element, dQ (not the total variation of energy, dU, used in the paper in defining tasis and posot), could be put into the form $q'dp$ where q' is of the dimensions of the velocity, and dp of the dimensions of momentum.

Dr. Donnan said that Mr. Swinburne's objections to entropy as a factor of heat energy seemed, at any rate as far as reversible operations were concerned, to be based either on a misconception, or on some novel conception of the real significance of such factors. All that was really known was that the "actions" of the external world on a given system could be expressed, in reversible thermodynamics, by differential terms of the form idm, where i and m were parameters of the system. In general, the separate terms were not perfect differentials, and the futility of an attempt to give a false reality to what were merely mathematical functions suitable under certain conditions had been pointed out by Planck. The suggestion to replace temperature by its square root so as to bring heat energy into line with electrical and kinetic energy had already been made by Mach. In dealing with irreversible actions the author claimed that the phenomena of heat conduction could be represented as a flux of posot along a down gradient of tasis according to the same law as a flux of quantity of electricity along a down gradient of potential. He pointed out that it was not necessary to abandon entropy to achieve this result, as Wiedeburg had shown that by making some very simple and plausible assumptions it was possible to bring entropy into line with other quantity factors, so that the entropy of a heat-isolated system remained constant, and the flow of heat could be represented as an entropy flux obeying a law analogous to that of Ohm.

Mr. Macfarlane Gray said that he had found the paper difficult to understand. Such expressions as:—"But the gas has increased its entropy by H/θ collected in the gas, and caught there," "the gas loses entropy," "a flow of entropy," were strange to him, his idea being that there is no entropy in any substance or in nature, neither is it a property or a quality of any body, but only length upon a diagrammatical representation of the related heat states of a body of unit mass, according to a systematised mental conception. He thought temperature and entropy were sufficient for this purpose, and he could see no advantage in the factors proposed. From certain statements in the paper he had concluded that chy was the specific heat of the working substance, but there were other statements inconsistent with this conclusion. He had, with what he had taken tasis to be, constructed a diagram to ascertain how the system proposed differed from the θ, ϕ, method. Starting with tasis proportional to the square root of the temperature he had not inquired what posot might be, because with heat energy and one ordinate fixed the other would declare itself. On this new diagram a graph is not a series of state points, and a cycle performed thermodynamically is not a closed curve. In the vapour field the adiabatics are sloped curves, and the dryness fraction is measured in the same way as on the old diagram. In the gas field he had set out problems similar to the illustrations in the paper, and found that the features were the same, so that he thought he had operated with the same "posot," although undisclosed.

Mr. Swinburne, in reply to Prof. Everett, said that τ and π were only proportional when the capacity was constant. The factors proposed were not only analogous to those used in electricity, but also to those used in other branches of physics. Prof. Bryan had discussed what ought to come in a subsequent paper, and evidently thought the subject should be commenced by dealing with generalised co-ordinates, whereas he (the author) had started simply, and on that account had used stand-pipes and perfect gases as illustrations. He remarked that although some members had found the paper difficult to follow, others had complained that it was too simple.

Mr. Eustace Large then exhibited some Twinned Crystals of Selenite, and the Society adjourned until February 14th.

Amylic Alcohol Formed during Fermentation.—G. Bemont.—Amylic alcohol formed during fermentation boils at 131°. It gives, on oxidation, an active valeric acid boiling at 175°, which seems to be the methylethylacetic acid. It probably only contains a little isopropylacetic acid.—*Comptes Rendus*, cxxxiii., No. 26.

NOTICES OF BOOKS.

Répertoire Général ou Dictionnaire Méthodique de Bibliographie des Industries Tinctoriales et des Industries annexes. Depuis les Origines j'usqu'à la fin de l'Année 1896. *Technologie et Chimie.* Par Jules Garçon. Ouvrage honoré du Grand Prix Décennal Daniel Dollfus, de la Société Industrielle de Mulhouse. Paris: Gauthier-Villars. 1900-1901. 3 vols. Roy. 8vo. Pp. 340 and 1638.

The descriptive title of this great "Bibliography of Dyeing and Related Industries" explains its character, but fails to convey any idea of the immense labour required to compile it, or to show its arrangement of matter. The latter point is the object of this notice.

The work begins with a statement of the principles controlling the admission of items and their classification; incidentally, we note that the author gives an account of the decimal system of classification orginating with Mr. Melvil Dewey (librarian of the Regents of the University, New York), and his reasons for rejecting the same. This introduction is followed by a chapter containing a list of the chemical bibliographies that have appeared in English, German, and French, and which serve as the sources of information used in this dictionary. The author gives the foremost place in chemical bibliography to Americans, and proves it by the long list of general and special works they have produced.

"Tableau I." contains a list of titles of subjects treated, in alphabetic order, from "*Accidents*" through "*Mordants*" to "*Zinc.*" "Tableau II." contains a list of the periodicals that were examined in preparation of the second and third volumes; they number 110, and comprise about 5000 individual volumes. After a list of abbreviations (Tableau III.) there is a long list of the proper names cited, and this occupies 263 pages, double columns; to each name is attached the numbers of the pages where they may be found; in this section there are no less than thirty-six references to the names A. G. and W. H. Perkin, and a score or more to J. J. Hummel.

The 1600 pages following contain the references, arranged first under subjects alphabetically, and secondly, under each subject by the periodicals examined; this economises space and type, yet with the copious indices makes the use of the work very simple. The titles of independent works precede the periodicals under each subject. Patent literature is included. Bibliographically the work is accurate.

It does not often fall to the lot of a book-reviewer to discover in a work of this character the elements of romance; it is a pleasure to note that the author, in thanking persons who assisted him in preparing the volume, includes the name of the young lady who laboured for months at arranging the slips. And he naïvely adds "now Mdme. Jules Garçon." He may be congratulated on the success of his enterprise in bibliography in at least two ways.

These volumes will be needed in the working libraries of all persons engaged in dyeing and colour-printing of textile fabrics, and they ought to be found for reference purposes in every public library. The three volumes are published at 100 francs. H.C.B.

Self-Examination for Medical Students. Third Edition, Enlarged. Philadelphia: P. Blakiston's Son and Co. 1901. Pp. 230.

This pocket-book contains 3500 questions on medical subjects arranged for self-examination. It also contains the proper references to standard works in which the correct replies will be found, as well as some examples of questions asked by the State Examining Boards of New York, Pennsylvania, and Illinois. As the practice of medicine is very much the same throughout the civilised world, this book will be useful to any English-speaking student. By its help the student can successfully question himself on all the important branches, or coach himself on any one subject in which he feels himself to be deficient. As a rule the questions have been selected with regard to their bearing on practical medicine, but at the same time there are throughout the book many unusual ones, thus giving the user a wide range of thought. We can recommend this book as being likely to be of great assistance to medical students, who would certainly qualify with ease if they had mastered only a part of the questions put.

A Text-book of Urine Analysis for Students and Practitioners of Medicine. By John H. Long, M.S., D.Sc. With numerous Illustrations. Easton, Pa.: The Chemical Publishing Company. Pp. 249.

The subject of practical urine analysis is treated in this volume in a concise manner, adapted to the requirements of medical students engaged in class-work, and also to the wants of the medical practitioner who has occasion to make more than the usual simple qualitative tests in urine examinations. A large number of quantitative methods are carefully described, and in such a manner that anyone with a previous training and experience in the manipulation of chemical apparatus should be able to follow them, while no doubtful methods have been included at all.

Numerous references are made throughout the text to the clinical significance of what is found by the various tests; and in the appendix a section is devoted to a tabular statement of the relation of pathological conditions to the chemical composition of the urine.

The book is divided into twelve chapters and an appendix; it is well and clearly printed and bound, and provided with a good index of ten columns. It should certainly be within the reach of, if not in the hands of, all medical students.

On the Composition of Dutch Butter. By Dr. J. J. L. van Ryn. London: Baillière, Tindall, and Cox. 1902. Pp. 48.

It has repeatedly happened, so the author tells us, that Dutch butter, the origin of which placed its genuineness and purity beyond doubt, has wrongly been declared by foreign (that is to say, not Dutch) chemists to be a mixture of butter and margarine. Great loss was suffered on this account, especially with regard to the English trade. The Dutch Government, therefore, caused a full examination to be made into the composition of Dutch butter produced in the latter months of the year, as it had been noticed that the difficulties arose almost exclusively in the course of each autumn. In order to become acquainted with the composition of Dutch butter, samples were analysed weekly from various parts of Holland, and particulars were collected under the following heads:—1. Number and breed of the cows from which the butter was obtained. 2. Age of the cows. 3. Date on which the cows last had a calf. 4. Approximate date of the next throwing of calf. 5. Nature of soil on which they grazed. 6. Nutrition. 7. Date of their being stabled.

The figures obtained by the examination of the butters have been arranged in order of time in twenty-five tables. These figures relate to refraction, specific weight, volatile acids, solid acids, dissoluble acids, saponification value, and iodine value.

From these figures it is seen that there are variations in the composition of the butters, and it was found that the rise in the percentage of volatile fatty acids begins shortly after the cows have been stabled. This is observed in all cases, and is very remarkable, because, until now, it has not been observed, or rather had escaped notice.

After describing a number of other experiments and generally discussing the question, the author states that he has established for a certainty that the abnormal chemical composition of Dutch butter during the autumn must be

attributed to the circumstance that the farmers leave their cattle in the meadows until late in the year, and he also claims to have proved that an earlier stabling prevents the calling into existence of the abnormal composition under discussion.

By "stabling," he points out, two varying factors are introduced; first, the cows are differently fed, and second, they are sheltered from the variations of weather and temperature. He has not, however, been able to decide with certainty whether both factors play an equal part, or whether one has a greater influence than the other.

Grundlinien der Anorganischen Chemie. By W. Ostwald. Leipzig: Wilhelm Engelmann. 1900.

In a lengthy preface to this admirable book the author explains his reasons for modifying in some respects the usual arrangement and order adopted in a text-book of inorganic chemistry, and on following his system throughout the volume, we feel that he is fully justified in his conclusions. The book represents the work of a teacher thoroughly conversant with the requirements of the student, and also of the fellow teacher, though in a lesser and subordinate degree. Recognising that, for various reasons, it is not possible to employ the ideal method, and build up chemistry as a rational science on the basis of certain principles, introducing the description of the various substances simply in illustration of these general laws, the author has fallen back on the plan of fitting in to the detailed description of the elements and their compounds, as opportunity offers, the discussion of the general principles and laws underlying the science, and has met with marked success, in spite of the special difficulties with which he has had to cope in the use of such a method.

Briefly, the chief features of the book are the strictly logical sequence in which ideas new to the student are put forward, and the lucidity of the explanations of theoretical portions of the subject which usually present great difficulty to the learner. Since the book only professes to deal with pure chemistry, practical applications receive but little attention, and manufacturing processes are only very shortly described.

CORRESPONDENCE.

THE VOLUMETRIC ESTIMATION OF MANGANESE.

To the Editor of the Chemical News.

Sir,—We do not agree with Mr. Ramage's summing-up of the issues between us; and with your permission we propose to make another and final attempt to show that for the general purposes of a steel works laboratory the use of ferrous sulphate is to be preferred to hydrogen peroxide.

The exception Mr. Ramage takes to the table of results published in our last letter, viz., "they are not of the high degree of accuracy established for our method," is an indication that he fails to grasp their significance. The figures obtained on the three steels by the bismuthate process as we prefer to work it were:—

0·31 1·01 0·89 per cent Mn.

The results obtained by separating the iron and manganese with acetate, &c., and finally making a bismuthate estimation (using hydrogen peroxide) on the pure manganese obtained from 2½ grms. of steel were:—

0·32 1·02 0·90 per cent Mn.

This latter process was devised out of regard for Mr. Ramage's distrust of any of the usual gravimetric processes, and we are prepared to take its indications as a standard. The variation between the two sets of figures just given is 0·01 per cent Mn. If one considers the widely different means by which these two sets of figures were reached, and the facts that the titrations were made with different solutions (H_2O_2 and $FeSO_4$), and that different weights of material were operated upon, the 0·01 per cent differences are very inconsiderable. Will Mr. Ramage notice that *under the best possible conditions* the variation in his results is only 0·004 per cent Mn less than in ours?

The following results which were obtained on the same steel, after destroying organic matter in the hot solution, show the comparative effects of excesses of hydrogen peroxide and ferrous ammonium sulphate in the titration of the filtered permanganatesolution:—

N/10 H_2O_2 used.	Mn obtained.	N/10 $(NH_4)_2(SO_4)_2Fe$ used.	Mn obtained.
C.c.	Per cent.	C.c.	Per cent.
5	0·497	5	0·490
10	0·530	10	0·494
15	0·577	15	0·493
20	0·605	20	0·490
25	0·655	25	0·485

This table shows:—

1. That accurate results can be obtained with H_2O_2 if *very* small excesses are used. We have never denied this.
2. That with increasing excesses of H_2O_2 the results are increasingly inaccurate. We contend that this is an undesirable feature in *any* reagent.
3. That accurate results are obtained by using ferrous sulphate in either small or large excess. This feature makes it superior to peroxide for *all* classes of steels, pig-irons, alloys, or any other ferriferous compound dealt with in steel works.

We trust the above statement will dispel Mr. Ramage's notion that during this discussion we have had in mind the assay of special steels only; we have desired only to emphasise the increased superiority of ferrous sulphate when dealing with such steels. Mr. Ramage's references to special steels are unfortunate. In his last letter he says:—"Hydrogen peroxide indicates the presence of the important rare metals in the special steels . . . . and special methods of analysis may be applied to them." It is not possible to detect molybdenum during the titration, nor is it possible to make an accurate estimation of Mn in a steel containing a few per cents Mo if hydrogen peroxide is used.

The question we raised respecting manganese alloys is certainly not answered by the paper in the *Transactions of the Chemical Society*, nor could it be because it depends entirely on facts which were not then publicly known. We have repeatedly titrated hydrogen peroxide against the permanganate in solutions containing 0·25 grm. of spiegel (after the Mn has been determined), and the amount of $KMnO_4$ required is always less than is required when the titration is made in acidified distilled water. If this difference is due, as we believe it is, to the reaction between ferric nitrate and hydrogen peroxide, surely Mr. Ramage ought to explain why, in the assay of alloys, he ignores the source of error he is so careful to avoid in steels.—We are, &c.,

Fred. Ibbotson.
Harry Brearley.

Bower Road, Sheffield.

Action of Propylic and Normal Butylic Alcohols on their Respective Sodium Derivatives; Synthesis of Dipropylic and Dibutylic Alcohols.—Marcel Guerbet.—Propylic and normal butylic alcohols, when heated to between 220° and 230° with their respective sodium derivatives, form condensed alcohols according to the equations:—

$$2C_3H_8O + C_3H_7NaO = C_6H_{14}O + C_3H_6NaO_2 + 4H,$$
$$2C_4H_{10}O + C_4H_9NaO = C_8H_{18}O + C_4H_7NaO_2 + 4H.$$

Further experiments prove this action to be general, at least for alcohols of the form R—CH_2—CH_2OH.—*Comptes Rendus*, cxxxiii., No. 26.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxiv., No. 1, January 6, 1902.

Preparation and Properties of Potassium Hydride.—Henri Moissan.—When potassium is kept for some hours in an atmosphere of hydrogen at a temperature of 350°, a transparent and crystalline layer of a hydrate is formed on the surface of the metal. This hydrate can be separated from the excess of metal by treatment with liquefied ammonia gas, in which case the metal is removed as potassium-ammonium. Analysis proves this hydride to have the formula KH. It is white and well crystalline, and instantly decomposed by water. It takes fire in cold fluorine, chlorine, and dry oxygen, and possesses very energetic reducing properties. It is comparable, both in appearance and chemical properties, to calcium hydride, which latter substance has been already described by the author.

Action of Cupric Hydrate on Aqueous Solutions of Metallic Salts.—A. Mailhe.—In a preceding paper, the author described the action of cupric hydrates on aqueous solutions of metallic chlorides and bromides. From this he was led to discover certain mixed basic salts analogous to those which he previously obtained by the action of oxides of mercury on aqueous solutions of salts of the same metals. He now describes the results obtained by the action of cupric hydrates on solutions of sulphates. The particular action examined is that of tetracupric hydrate, the only one which gives well crystalline and well defined compounds. Péligot's blue hydrate always leads to an uninteresting amorphous basic salt. The special salts prepared were—

$2SO_4Cd,3CuO.12H_2O$,
$2SO_4Cd,3CuO.10H_2O$,
$2SO_4Ni.3CuO.12H_2O$,
$2SO_4Ni.3CuO.10H_2O$,
$3SO_4Zn.5CuO.16H_2O$,
$2SO_4Co.3CuO.10H_2O$,
$SO_4Zn,3CuO.5H_2O$,
$2SO_4Zn,3CuO.12H_2O$,
$2SO_4Cu.3ZnO.12H_2O$,
$SO_4Zn.2CuO.5H_2O$,
$2SO_4.3CuO.12H_2O$,
$SO_4Zn.3CuO.5H_2O$,
$2SO_4Zn.3CuO.12H_2O$.

Condensation of Acetylenic Carbides with the Ether Salts. Syntheses of Acetylenic Acetones and β-Ketonic Ethers.—Ch. Moureu and R. Delange.—The authors show that formic ethers attack sodium acetylenic carbides energetically, and that the action of water on the product of the reaction engenders the acetylenic aldehydes, R—C≡C—CHO. This reaction is, however, a complex one, and in nearly all cases the β-ketonic ethers corresponding to the ether salts employed are produced. Experiments were performed, using very varying proportions of two acetylenic carbides, œnanthylidene (C_5H_{11}—C≡CH) and phenylacetylene (C_6H_5—C≡CH), and on ten ether salts. The authors also devise a theory by which the reaction may be explained.

MISCELLANEOUS.

Direct Determination of the Avidity of Ammonia and of the Amines.—D. Konovalof.—The method used is based on the measurement of the tension of the free bases in solution. The conditions are most favourable when one of the bases under comparison is not volatile at the temperature of the experiment (60°). The calculation is very simple in the case of weak solutions where the presence of salts has only a negligible influence on the tension, which may be looked upon as proportional to the concentration. Under these conditions, exact values can be obtained of the reciprocal displacement of the amines by means of the formula $H'x+H''(1-x)=H$; where H' and H'' represent the tensions of the two amines in such cases where they are entirely in the free state in solution, and H the tension observed in a mixture of the salt of an amine with an equivalent amount of the other amine; x is the proportion displaced of one of the amines. The experiments were carried out with the chlorhydrates of ammonium, benzylamine, methylamine, dimethylamine, trimethylamine, and triethylamine. For the relative values of their avidity the figures correspond fairly well with their electric conductivity, but are not strictly proportional. With 0·5 normal concentrations the following values were found :—

Ammonia	1·00
$C_6H_5.CH_2.NH_2$	0·97
CH_3NH_2	4·32
$(CH_3)_2NH$	3·90
$(CH_3)_3N$	2·21
$(C_2H_5)_3N$	1·68

These figures have no relation to the heats of neutralisation; thus, $(CH_3)_3N$ displaces 70 per cent of benzylamine from its salt, while the heats of neutralisation are 8·9 cal. for the first, and 12·9 cal. for the second.—*Journ. Soc. Phys. Chim. R.*, vol. xxxii., p. 193.

NOTES AND QUERIES.

Ammonium Chloride and Oxidising Agents.—I happened to heat together some ammonium chloride and sodium nitrate a short time ago, and found—rather to my surprise—that chlorine was given off in rather large quantities. I have since tried the effect of heating ammonium chloride with other oxidising agents, and find that chlorine is given off with potassium nitrate and potassium chlorate, but that ammonia is produced with manganese dioxide, mercuric oxide, and lead dioxide; the amounts of gases given off seem to vary with the temperature, thus causing an accurate quantitative estimation of the reaction to be difficult. I can find no mention of the reaction in any text-book, so should be much obliged if you could give me some information as to what happens in your "Notes and Queries," or refer me to a text-book in which I could find it.—F. W. Hodges.

MEETINGS FOR THE WEEK.

Monday, 3rd.—Society of Arts, 8. (Cantor Lectures). "The Purification and Sterilisation of Water," by Samuel Rideal, D.Sc., F.I.C.
—— Royal Institution, 5. General Monthly Meeting.
Tuesday, 4th.—Royal Institution, 3. "The Cell—its means of Offence and Defence—Immunity," by Allen Macfadyen, M.D., B.Sc.
—— Society of Arts, 4.30. "The History of the Rosary in all Countries," by the Rev. Herbert Thurston, S.J.
Wednesday, 5th.—Society of Arts, 8. "Jamaica," by Herbert T. Thomas.
Thursday, 6th.—Royal Institution, 3. "The Scot of the Eighteenth Century—I., At Home," by the Rev. John Watson, D.D. (Ian Maclaren).
—— Society of Arts, 4.30. "The Coal Resources of India," by Wyndham R. Dunstan, F.R.S.
—— Chemical, 8. "An Investigation into the Composition of Brittle Platinum," by W. N. Hartley. "Conversion of *l*-Hydroxycamphene into β-Halogen Derivatives of Camphor," by M. O. Forster. "Tetrazoline" (Part II.), by S. Ruhemann and H. E. Stapleton. "The Solubilities of the Calcium Salts of the Acids of the Acetic Acid Series" and "The Equilibrium between a Solid and its Saturated Solution at various Temperatures," by J. S. Lumsden. "The Influence of Temperature on Association in Benzene Solution, and the Value of the Molecular Rise of Boiling-point for Benzene at different Temperatures," by W. R. Innes. "The Magnetic Rotation of Ring Compounds—Camphor, Limonene, Carvene, Pinene, and some of their Derivatives," by W. H. Perkin, sen., F.R.S. "Polymerisation Products from Diazoacetic Ester," by O. Silberrad.
Friday, 7th.—Royal Institution, 9. "The New Mammal from Central Africa and other Giraffe-like Animals," by Prof. E. Ray Lankester, M.A., LL.D., F.R.S.
Saturday, 8th.—Royal Institution, 3. "Landmarks in the History of Opera—IV., Wagner" (with Musical Illustrations), by W. H. Hadow, M.A., Mus Bac.

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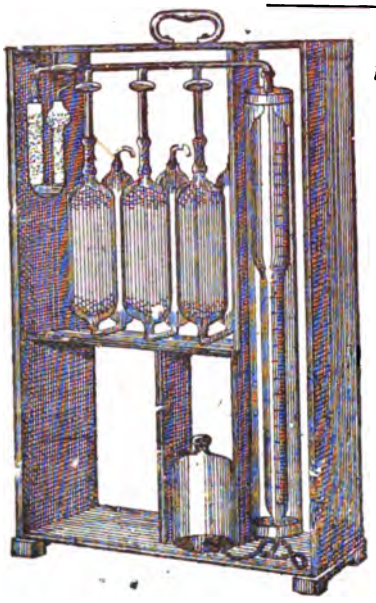

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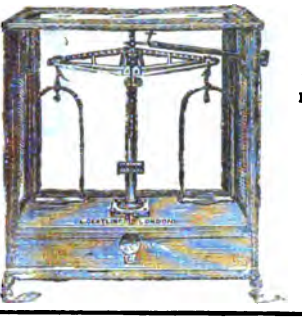

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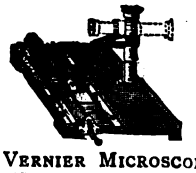

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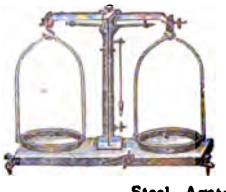

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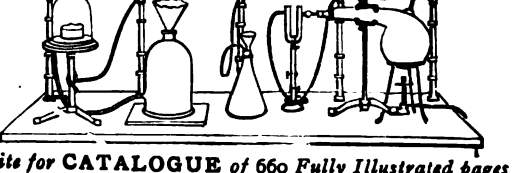

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THE CHEMICAL NEWS.

VOL. LXXXV., No. 2202.

RESEARCHES ON THE PRESSURE FORCES OF LIGHT.*

By PETER LEBEDEW.

(Concluded from p. 54).

The following method was adopted for the measurements with the first calorimeter (Fig. 6). The mirror apparatus (Fig. 1) was drawn back so far on the sliding scale that the vane apparatus could be replaced by the calorimeter diaphragm, D. The calorimeter was then illuminated for five minutes, and from minute to minute the thermometer readings (and in the intervals the galvanometer readings also) were taken. Then the rays were stopped by a screen, and during the next five minutes, from minute to minute, the fall of the thermometer (and the zero point of the galvanometer) observed. A complete series of observations included five successive periods of illumination.

All observations were graphically represented, the readings of the thermometer being marked on squared paper and united by as even a curve as possible (Fig. 9).

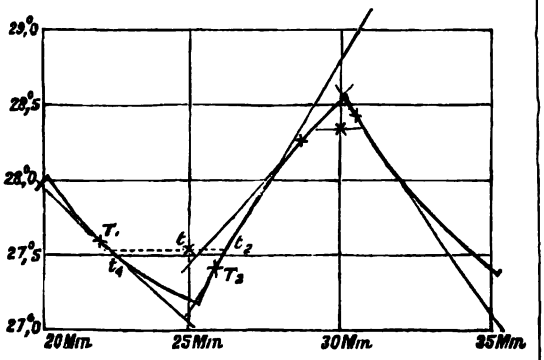


FIG. 9.

It is clear that after 10 secs. the movement of the mercury in the thermometer shows by a turning point when the light was turned on or off.

The great rapidity with which the calorimeter cools must be specially allowed for, for even in the interval of an observation neither the rate of warming nor of cooling is constant. For definite mean temperature of the *surface* of the calorimeter both rates have constant values, which, as is easily seen from the figure, are represented by tangents; for these constant values the points at which the tangents cut the ordinates give the temperature differences which would be reached in five minutes if both rates were constant, and the sum of both differences gives the total corrected rise of temperature.

But here an error in the estimation of true mean temperature may appear: the thermometer remains a little below this temperature in that it marks a lower temperature when the light is turned on, and a higher when the light is turned off. The fact that the thermometer marks a turning point after 10 secs. gives as a first approximation (and a sufficiently close one for us) the assumption that the thermometer is 20 secs. behind time. For a surface temperature, t, the points to be compared in the curve would be not t_1 t_2, but those dating 20 secs. back, *i.e.*, T_1 and T_2.

Such graphic determinations were made for two thermometers. Table III. gives a series of measurements.

TABLE III.

Calorimeter I. Water Equivalent 3·13 grms.

	Rate of warming.	Rate of cooling.	Total warming.	Galvanometer deflection.	Reduced total warming. G=100.
	°	°	°	Divs.	°
I.	1·67	0·63	2·30	140	1·64
	1·49	0·80	2·29	128	1·79
II.	1·44	0·85	2·29	128	1·79
	1·31	1·10	2·40	122	1·97
III.	1·38	1·08	2·46	129	1·91
	1·00	1·37	2·37	126	1·88
IV.	1·30	1·15	2·45	123	1·99
	1·04	1·45	2·49	127	1·96
V.	1·26	1·27	2·54	129	1·97
	0·93	1·50	2·43	126	1·93

Total warming in five minutes (G=100) 1·88 ± 0·09°.

The measurements were much simpler with the second calorimeter. The calorimeter was cooled with ethyl ether, then the light was turned on it, and the thermometer read from minute to minute (in the meanwhile the temperature of the bath and the galvanometer were observed). The results were represented graphically, and the point was marked at which the temperature of the calorimeter coincides with that of the bath (taking into consideration that the thermometer was 20 secs. behind time), and the tangents of the true rates of warming drawn at these points, and also the difference of the thermometer readings at intervals of 2·5 mins. Such a series of readings was taken three times; Table IV. gives the results of measurements.*

TABLE IV.

Calorimeter II. Water Equivalent 3·61 grms.

	Rise of temperature in five minutes.		Mean value.	Galvanometer deflection.	Reduced rise of temperature. G=100.
	Tangents.	Differences.			
	°	°	°	Divs.	°
I.	2·40	2·41	2·40	159	1·51
II.	2·55	2·57	2·56	163	1·57
III.	2·43	2·50	2·46	158	1·56

Mean rise of temperature in five minutes (G=100) 1·55 ± 0·02°.

Hence, the quantity of energy per sec.—

$$E = \frac{1{\cdot}55^\circ \cdot 3{\cdot}61 \cdot 4\,18 \cdot 10^7}{300} = 7{\cdot}74 \cdot 10^5 \text{ ergs.}$$

In our experiments the incident rays were not parallel but convergent; the small inclination of the incident rays would render a correction (of about 1 per cent) necessary; this correction balances the other errors in the measurements, and may be neglected. The calculation is sufficiently accurate if we employ the formula which holds good according to Maxwell and Bartoli for a beam of parallel rays.

For an absolutely black body the pressure based on the values of Table IV. is—

$$p = \frac{E \text{ (ergs)}}{3 \cdot 10^{10}} = 0{\cdot}0000258 \text{ dynes.}$$

In order to express the results in such a way as to be readily compared at a glance, the value of Maxwell and Bartoli's pressure for an absolutely black body calculated from the calorimeter measurements was adopted as an arbitrary unit; this unit was called an M.B. unit.

* From the *Annalen der Physik*, 1901, vol. vi., p. 433.

* The results of Tables III. and IV. cannot be directly compared with one another since they refer to different adjustments of the thermo battery.

RESULTS.

	Apparatus I.	Apparatus II.							Apparatus III.	
		Calorimeter I.			Calorimeter II.					
		White.	White.		White.	Red.	White.	Blue.	White.	
1. Thick platinum vane	1·8 ± 0·2	1·6 ± 0·1	1·5 ± 0·1	—	—	—	—	—	1·5 ± 0·1	1·4 ± 0·1
2. Thin platinum vane.	1·3 ± 0·2	—	—	—	—	—	—	—	1·2 ± 0·1	1·1 ± 0·1
Calculated	1·2	—	—	—	—	—	—	—	1·1	1·0
3. Platinum, thick ..	—	1·8 ± 0·1	—	—	—	—	—	—	—	—
4. Platinum, thin ..	—	2·0 ± 0·1	1·9 ± 0·2	1·8 ± 0·1	1·9 ± 0·1	(1·8 ± 0·8)	1·7 ± 0·1	(1·5 ± 0·5)	1·7 ± 0·1	2·0 ± 0·1
5. Aluminium, thick ..	—	—	2·3 ± 0·4	1·9 ± 0·1	—	—	—	—	—	—
6. Aluminium, thin ..	—	—	2·0 ± 0·1	2·3 ± 0·1	2·0 ± 0·2	(2·9 ± 0·8)	2·1 ± 0·1	(2·5 ± 0·5)	1·4 ± 0·2	1·7 ± 0·1
7. Nickel, thin	—	—	1·7 ± 0·3	1·2 ± 0·2	1·4 ± 0·1	(2·3 ± 0·5)	1·4 ± 0·2	(2·7 ± 0·9)	—	—
8. Mica	—	—	—	—	—	—	—	—	0·08 ± 0·05	0·13 ± 0·03

Expressed in these units, the result of Table I. is—

$$p = \frac{0{\cdot}0000308 \pm 0{\cdot}0000017}{0{\cdot}0000258} = (1{\cdot}19 \pm 0{\cdot}07)\ \text{M.B.}$$

A direct measure of the reflecting power of the vane used was rendered useless owing to the inequalities of the thin metal leaf. Therefore, I observed with the photometer (Fig. 8) the reflecting power of the leaf of which the vanes were made. With this also the inequalities produced strong effects, and also a distinct colouration of the reflected light was apparent (especially in the case of nickel). The reflecting power observed for an angle of incidence of 25° is therefore given in Table V. without further reductions, and hence are calculated in M.B. units the pressure forces to be expected. For the sake of comparison the reflecting power according to Hagen and Rubens of a perfectly reflecting surface for normal incidence ($\lambda = 600\ \mu\mu$) is given, and also the pressure forces calculated from this reflecting power. (The numbers for magnalium are given under aluminium).

TABLE V.

	Photometric measurements.		According to Hagen and Rubens.	
	ρ.	p. M.B.	ρ.	p. M.B.
Platinum ..	0·5 ± 0·05	1·5	0·64	1·64
Aluminium ..	0·6 ± 0·05	1·6	0·83	1·83
Nickel	0·35 ± 0·05	1·4	0·65	1·65

I have omitted the calculations for mica because the observations were only made with one vane, and a control experiment with a thicker vane failed.

The results of the single series of experiments with the three kinds of vane apparatus are given in the accompanying table. When I proceeded to take measurements at lower temperatures, since the results at the temperature of the room were uncertain and fluctuating, I did not hope to attain in the experiments such a great degree of agreement with the calculations of Maxwell and Bartoli as is shown in the first experiments with Apparatus II. I believed at first that this agreement was accidental, and therefore replaced Calorimeter I. by Calorimeter II., and then Apparatus II. by Apparatus III.

The numerous observations which I made with Apparatus I. at the temperature of the room are decidedly less satisfactory than the later measurements, and are therefore not given. Also, the measurements with vane 2 of Apparatus II. are omitted, because a supplementary microscopic examination showed insufficient platinisation. Unfortunately, with Apparatus III. only two series of measurements were taken because the arrangement was then destroyed by an accident.

The results are expressed in M.B. units; under each value is also given the mean variation of the position of the vane apparatus in the same units, all variations amounting to less than 0·15 M.B. being denoted by 0·1, and all under 0·25 by 0·2.

In estimating the accuracy of the final results the following considerations must be taken into account:—The variations of the deflection are given in the table; the magnitude of the pressure resulting from the deflection in absolute measure (direction force of the thread, distance of the scale, lever of the vanes, and corrections for the illuminated surfaces) may be determined with an accuracy of ±8 per cent, the calculation of the absolute value of the M.B. unit from calorimeter measurements (water equivalent, rise of temperature of the calorimeter, and the ratio approximately equal to unity of the diaphragm to the vane) is accurate to ±7 per cent; the error in the determination of the reflecting power may reach about 10 per cent. These errors in the measurements are exceeded by the inaccuracies in placing the vane in the centre of the real image of the diaphragm, and the possibility that the heat radiation of an illuminated vane is concentrated by the surface of the globe on other parts of the vane apparatus, and that the amount of heating alters at each oscillation of the vane apparatus. For white light the still admissible error of the measurement may be estimated at about ±20 per cent.*

In experiments with blue and red light, where the available energy of radiation reaches scarcely one-fifth of that of white light, the disturbances caused by convection are the same, and therefore the errors correspondingly greater. The same holds good for the very small deflections (scarcely reaching 4 div. double deflection) in the case of mica. These control experiments are sufficient to confirm the fact that in these cases also no new effect of light showed itself, which would be comparable with the forces of Maxwell and Bartoli.

* A refinement of the methods of measurements would only essentially increase the certainty of the results if the arc lamp is replaced by another constant source of light.

I made, moreover, many comparative experiments with thick and thin platinum and aluminium vanes, but I did not succeed in establishing with certainty a perceptible radiometer difference; therefore within the limits of errors of observation the radiometer effect in the case of thin metal vanes is to be regarded as zero.

The results obtained may be collectively expressed as follows:—

1. An incident beam of light exercises a pressure both on an absorbing and a reflecting body. This force capable of doing work ("weight-moving") is dependent upon Crookes's already known secondary forces caused by heating, and upon the phenomena of convection.

2. These pressure forces of light are directly proportional to the quantity of energy falling on the body, and are independent of the colour of the light.

3. These pressure forces of light agree quantitatively within the limits of errors in the experiments with the "weight-moving" forces of radiation calculated by Maxwell and Bartoli.

Thus, the existence of Maxwell and Bartoli's pressure forces of light beams is experimentally proved.

ON PYRITE AND MARCASITE.*

By H. N. STOKES.

(Continued from p. 43).

III. Details of the Method.

Necessary Precautions.

In the following the precautions necessary to obtain strictly concordant results are given in detail, as well as a statement of the abbreviations which are permissible when only approximate results are desired.

To obtain accurate results it is necessary—

1. To employ sulphide absolutely free from oxidation products.
2. To avoid all access of free oxygen during the experiment.
3. To prevent the precipitation of basic ferric sulphate.
4. To prevent change of concentration due to loss by evaporation.
5. To make the titration with the greatest possible accuracy.

Preparation of the Material.

The mineral is somewhat finely ground, an operation which is greatly facilitated by adding water. After drying, it should be kept in a vacuum or in carbon dioxide until used. As both pyrite and marcasite oxidise rapidly when powdered, the oxidation products must be completely removed immediately before the experiment. About 1 grm. of the dry powder is washed by decantation two or three times with ether, two or three times with 20 per cent hydrochloric acid (1 part concentrated and 1 part water), and heated with the latter for a quarter of an hour or more, to remove basic salts, oxides of iron, and any contaminating sulphides or soluble silicates which may thus be extracted. It should be remarked that under these conditions pure pyrite evolves no perceptible hydrogen sulphide† if tested a few moments with lead-paper, while marcasite continues to give off minute amounts however long it may be heated with the acid. The evolution of hydrogen sulphide from pyrite indicates the presence of other sulphides. The material is then collected on a Gooch crucible fitted with a disk of Schleicher and Schüll hardened filter-paper, and washed with dilute hydrochloric acid, and then with water which has been boiled and cooled in carbon dioxide. The washing is conducted in an atmosphere of carbon dioxide, and for this purpose I use the apparatus shown in Fig. 1, in which the wash-water is delivered from the dropping funnel, and the carbon dioxide, supplied from an ordinary automatic generator,* itself affords the pressure necessary to drive the liquid through the filter. The crucible is then sucked out by attaching a pump to the outlet tube, placed for a moment on a blotter to remove most of the water, and transferred to a vacuum desiccator containing sulphuric acid, and filled with carbon dioxide, which is then exhausted. The use of carbon dioxide in this connection is essential, as even the best vacuum attainable by ordinary means contains enough oxygen to produce sensible oxidation. The drying is complete in half-an-hour.

Oxidation Apparatus.

The decomposition apparatus is represented in Fig. 2. A flask holding about 400 c.m.³ is provided with a doubly perforated rubber stopper which has been well boiled out in caustic soda; through this pass the inlet tube for carbon dioxide and the condenser. The latter consists of an elongated calcium chloride tube, into which fits loosely a test-tube through which cold water passes. The condenser has a hole at *a* to allow the passage of the steam, so that the return water drops quietly from the end of the tube. It is absolutely essential to prevent the return water from striking the sides of the flask or inlet tube, as at such points a film of basic sulphate invariably forms.

The flask is placed up to the stopper in a cylinder of asbestos board provided with windows of mica or glass on opposite sides, to admit of watching the operation. This rests on a plate† of asbestos containing a circular hole covered with gauze, and is closed at the top by a similar plate. The object of this arrangement is to prevent as far as possible the condensation of water on the sides of the flask; when this occurs, a ring of basic sulphate tends to form at the surface of the liquid. About 250 c.m.³ of the standard solution are used. The small loss by wetting of the condenser is compensated by having it wet at the outset.

Blank experiments of this apparatus lasting two hours show no deposition of basic salt, no loss by volatilisation (if the ingoing gas is saturated with moisture), and no reduction of the ferric salt.

Details of the Operation.

As the solution contains enough air seriously to affect the results, and as the percentage of sulphur oxidised varies with the temperature, it is necessary to introduce the substance into the boiling liquid, and not until all the air has been boiled out. For this purpose it is rapidly placed in the small bucket, shown on a large scale in Fig. 2, A, which consists of a small test-tube cut off and constricted at the bottom, and provided with a loosely fitting platinum disk, made tight by pouring in a little iron-free asbestos emulsion, such as is used in Gooch crucibles. The bucket is suspended by a platinum wire, and is dropped into the liquid after this has boiled ten minutes. It is necessary that the powder shall disseminate through the liquid, and not settle out on the bottom for any considerable time. If this be not the case, abnormal results are obtained, for reasons which are not clear. The powder, as well as the apparatus, must therefore be strictly free from grease, and, to aid the dissemination, the apparatus must be so mounted on a stand as to admit of agitating the liquid until the powder remains suspended. Pyrite disseminates readily, but marcasite shows a strong tendency to flocculate at first, and the two may generally be distinguished by this means.

During the whole operation a very slow current of carbon dioxide is passed through. This must, of course,

* From the *Bulletin of the U.S. Geological Survey*, No. 186.

† One-half grm. pure powdered pyrite, boiled in the decomposing apparatus for one hour with 7 per cent sulphuric acid, in a slow current of carbon dioxide, caused a slight blackening of lead paper placed in the top of the condenser.

* Liquefied carbon dioxide contains air, and should not be employed.

† In the figure the flask is represented as elevated above the plate. During the boiling it must rest on it.

be absolutely free from oxygen, and as some air is present even in the most carefully prepared carbon dioxide, I always pass it over red-hot copper.* It must also be free from hydrogen sulphide, which is not always the case, and it can easily be freed from this by passing it through a tube filled with beads, and containing saturated copper sulphate solution.

The greater part of the ferric sulphate is reduced after a few minutes' boiling, but in order to effect complete reduction the boiling should be continued two hours. The liquid is then cooled and filtered through dry filters into two accurate 100 c.m.³ flasks, which are filled with carbon dioxide, and placed in cylinders of the same. In order entirely to avoid contact with the air, it is driven over

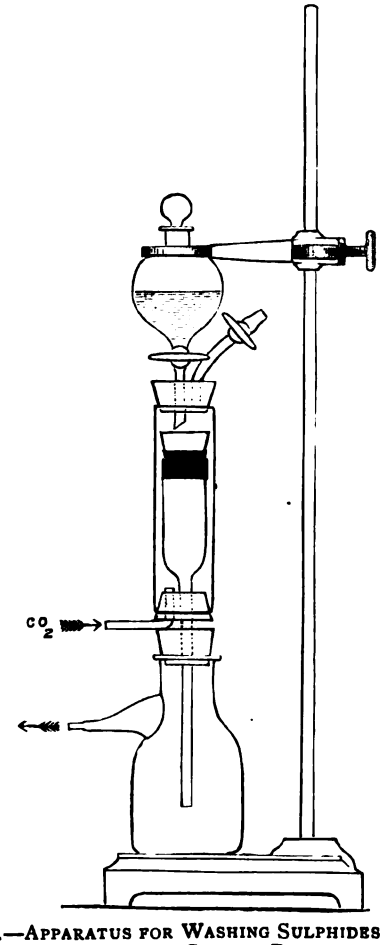


FIG. 1.—APPARATUS FOR WASHING SULPHIDES IN AN ATMOSPHERE OF CARBON DIOXIDE.

through the inlet tube by means of carbon dioxide introduced through the condenser. The flasks are then brought to the temperature of the permanganate to be used, and the contents are transferred to flasks containing sulphuric acid, and filled with carbon dioxide, and titrated. After reducing by hydrogen sulphide, filtering off the sulphur, and expelling the hydrogen sulphide by boiling in a carbon dioxide current, the total iron is titrated. A portion of the remaining solution should always be tested with sulphocyanate to ascertain the extent of the reduction. This generally gives a faint reaction for ferric iron If it is decidedly red, either the reduction is incomplete or the mineral contains notable amounts of copper. (For the explanation of this see "Experiment with Ferrous and Cuprous Sulphates," *prox.*).

As small errors in titration produce considerable deviation, an accurately calibrated burette should be used, so narrow that the bottom of the meniscus can be distinctly seen against a light. Those burettes which have the graduation carried half-way around the tube are particularly good for accurate reading. The permanganate should have a strength of about 1·5 grms. per litre, and should be run in only to the very faintest change of colour, as compared with a ferric alum solution of approximately the same strength and acidity.

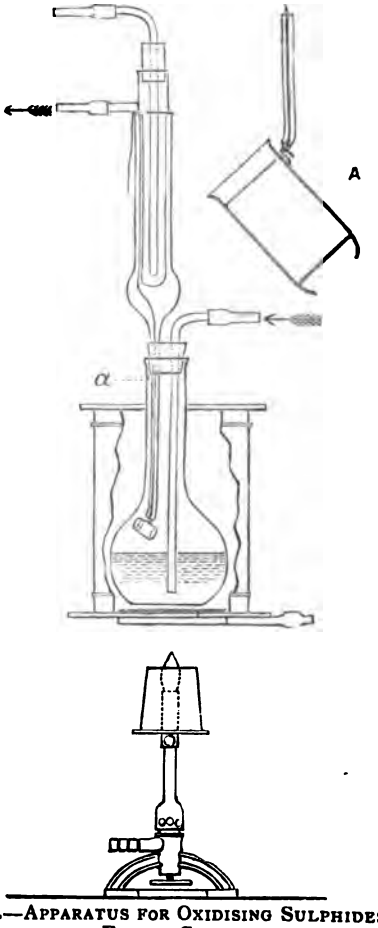


FIG. 2.—APPARATUS FOR OXIDISING SULPHIDES WITH FERRIC SOLUTION.

When less accurate results are required the mineral, after extraction with acid, may be simply collected on a hardened filter, washed with acid and water, and dried in the vacuum; special purification of the carbon dioxide, filtering in a carbon dioxide atmosphere, filtration of the reduced solution from sulphur, and other exceptional precautions may be omitted. Results made in duplicate in this way may differ 3 or 4 per cent.

Accuracy of the Method.

If 100 c.m.³ solution and a permanganate of 1·5 grms. per litre be used in titrating an excess of 0·1 c.m.³ permanganate, or an oxidation of 0·0003 grm. of the material, gives the following errors in the value of p :—

* When many determinations are to be made, it is convenient to place the copper in an iron gas-pipe, water jacketted at both ends if necessary.

	a.	b.	c.	Oxidation of 0·0003 grm. sulphide.
Pyrite ..	+1·00	+0·12	−1·00	−1·00
Marcasite	+0·20	+0·05	−0·20	−0·15

Special care is therefore necessary in standardising the original solution and in the final titration. Practically, duplicate determinations with pyrite tend to agree more closely than those with marcasite, as seen in the table below. This is perhaps due to the greater tendency of marcasite to flocculate, as indicated above, and to its greater tendency to oxidise, although the influence of the latter is less than in the case of pyrite. The influence of various impurities is considered later on.

(To be continued).

EXPERIMENTS RELATIVE TO THE CONSTITUTION OF PECTOLITE, PYROPHYLLITE, CALAMINE, AND ANALCITE.*

By F. W. CLARKE and GEORGE STEIGER.

(Continued from p. 55).

Analcite.

ANALCITE, from many points of view, is a species of peculiar interest, and of late years it has received a great deal of attention. Its formula may be written in various ways, especially as regards the interpretation of its one molecule of water; but evidence too often has yielded before preconceived opinion. Additional evidence is now available, partly from the experiments of Friedel and partly from the data obtained during the present investigation.

The analcite examined by us was in well developed crystals from Wasson's Bluff in Nova Scotia. A uniform sample was prepared as usual, and the analysis given below is contrasted with the theoretical composition required by the accepted empirical formula $NaAlSi_2O_6,H_2O$.

Analysis.

	Found.	Calculated.
SiO_2	57·06	54·55
Al_2O_3	21·48	23·18
Fe_2O_3	0·13	—
CaO	0·16	—
Na_2O	12·20	14·09
H_2O at 100°	0·58	—
H_2O over 100°	8·38	8·18
	99·99	100·00

Fractional Water.

At 100°	0·58
At 180°	1·16
At 260°	3·64
At 300°	1·57
Low redness	1·90
Full redness	0·11
Blast	none
	8·96

The fractional water determinations were made by heating in an air-bath to constant weight at each temperature up to 300°, and finally over the direct flame. The first fraction, at 100°, is evidently hygroscopic or extraneous water, which can be disregarded. The remainder of the water, 8·38 per cent, belongs to the species. The significance of the analytical figures will be considered later.

Upon boiling the powdered analcite with sodium carbonate solution—250 grms. to the litre, as in all the preceding experiments—0·73 per cent of silica was extracted. After ignition, the mineral in two determinations yielded 1·46 and 1·38 per cent respectively. The splitting off of silica is therefore very slight; and one of the formulæ proposed by Doelter (*Neues Jahrb. für Min.*, 1890, vol. i., p. 133), $Na_2Al_2Si_2O_8+2H_2SiO_3$, may be set aside as improbable. Metasilicic acid or an acid metasilicate can hardly be present in analcite, although the possibility of a neutral metasilicate, as indicated by the empirical formula, is not excluded. By Doelter's formula one-half of the silica ought to be removable.

Upon heating analcite with dry ammonium chloride, results of a remarkable character were obtained. Sodium chloride was formed which could be leached out by water and measured, while ammonia free from chlorine was retained by the residue to a notable and surprisingly stable degree. The experiments in detail were as follows:—

A. Analcite, mixed with four times its weight of ammonium chloride was heated for four hours to 350°. There was a gain in weight of 2·18 per cent, and 6·10 per cent of soda, or one-half of the total amount, was converted into NaCl, which was leached out by water, examined as to its purity, and weighed. In the residue 1·20 per cent of silica was extractable by sodium carbonate, showing that no more splitting off had occurred than was previously observed. The gain in weight, as will be seen from subsequent experiments, is due to the fact that all of the NH_4Cl had not been driven off; or else that more water was retained.

B. Analcite was ground up with four times its weight of NH_4Cl, heated for several hours, re-ground with another fourfold portion of chloride, and heated to 350° for twenty-one hours. Gain in weight, 0·08 per cent; 5·57 per cent of soda was extracted as chloride.

C. Analcite heated to 350° for eight hours, with four times its weight of NH_4Cl. Loss of weight, 0·10 per cent.

D. Six grms. of mineral and 28 of chloride, mixed by thorough grinding, were heated to 350° for fourteen hours; then were re-ground with 28 grms. of fresh NH_4Cl and heated for thirty-five hours. Loss of weight, 0·13 per cent; 5·07 per cent of soda was extracted as chloride, plus 0·14 of ammonium chloride unexpelled; 2·03 per cent of silica was rendered soluble in sodium carbonate.

So far, three facts are noticeable. First, the weight of the mineral after treatment is almost exactly the same as before; showing that gains and losses have balanced each other. Second, little silica has been split off. Third, approximately—but not rigorously—one-half of the soda had been converted into NaCl. In A, it was exactly half; in the other experiments, a little less than half. Furthermore, in the sodium chloride dissolved out there is only a very little ammonium chloride, amounting at most to 0·14 per cent, calculated upon the weight of the original mineral.

In the residue of the analcite after extraction of sodium chloride, abundant ammonia can be detected, with either no chlorine or at most a doubtful trace. If, however, the unleached mineral, still retaining its sodium chloride, be heated strongly, say from 400° up to redness, NH_4Cl is re-generated and given off. Its absence, as such, both from the leach and the residue was repeatedly proved. The ammonia and water retained by the analcite after heating to 350° with ammonium chloride were several times determined, and the following percentages, still reckoned on the original mineral, were found:—

	NH_3.	H_2O.
In B	2·03	2·25
In C	2·19	2·00
In D	2·36	1·89
In D	2·35	—
In D	2·06	—
Mean	2·20	2·04

* *Bulletin of the United States Geological Survey*, No. 167.

Correcting the ammonia for the 0·14 of NH_4Cl found in D, the mean value becomes 2·15. This permanent ammonia is not given off upon warming the material with caustic soda solution, and is therefore not present as a salt. The determinations of it were made by three distinct methods, and there is no possible doubt as to its presence and character.

The composition of the analcite after the treatment with ammonium chloride may now be considered, with the subjoined combination of the data. The NaCl in A, 11·50 per cent, was in material which had gained 2·18 per cent, and is subject to a correction which reduces the figure to 11·26. In B, C, and D the corresponding correction is so small that it may be neglected. The last column gives the composition of the leached residue, re-calculated to 100 per cent, after deduction of NaCl and the soluble silica. The letters refer back to the several experiments, and the little iron is included with the alumina.

	A.	B.	C.	D.	Average.	Residue.
Soluble SiO_2 ..	1·20	—	—	2·03	1·61	—
Insoluble SiO_2 .	—	—	—	54·96	54·96	62·59
Al_2O_3	—	—	—	21·37	21·37	24·34
CaO	—	—	—	0·16	0·16	0·18
NaCl	11·26	10·50	—	9·57	10·44	—
Na_2O	—	—	—	7·12	7·12	8·11
NH_3	—	2·03	2·19	2·21	2·15	2·46
H_2O	—	2·25	2·00	1·89	2·04	2·32
				99·31	99·85	100·00

The analcite residue, like the original mineral, is completely decomposable by aqueous hydrochloric acid. It may be a mixture, but it seems more probable that it is a definite compound, for it approximates in composition to the formula $H_2Na_2Al_4Si_8O_{24}.NH_3$.

This represents a quadrupled analcite formula, in which half of the sodium is replaced by hydrogen, and with NH_3 in place of $4H_2O$. The analytical comparison is as follows:—

	Found.	Calculated.
SiO_2	62·59	61·46
Al_2O_3	24·34	26·12
CaO	0·18	—
Na_2O	8·11	7·94
H_2O	2·32	2·30
NH_3	2·46	2·18
	100·00	100·00

The agreement is not close, but it is close enough to be suggestive and to indicate the character of the reaction which has taken place. It needs, however, verification by additional experiments upon other preparations, and upon analcite from other sources. In this connection it may be well to reiterate that the substance was prepared by very long heating at 350°, and is therefore stable at that temperature.

An interesting feature of these experiments is their harmony with the researches of G. Friedel (*Bull. Soc. Min.*, 1896, vol. xix., p. 94), who has shown that the water of zeolites may be replaced by ammonia and other substances, without change of the crystalline structure. In the case of analcite, ammonia was taken up to the extent of 2·04 per cent, or almost exactly the amount found in our analcite residue. The great difference between Friedel's method of experimentation and ours renders the agreement all the more striking, and sustains our belief that the mineral and the residue are compounds of the same general order. We hope to continue our experiments and to extend our investigations to other zeolites.

If, now, analcite and its derivative, our analcite residue, are analogous compounds, the relation between them is expressed by these formulæ:—

Analcite .. .. $Na_4Al_4Si_8O_{24}.4H_2O$.
Analcite residue .. .. $H_2Na_2Al_4Si_8O_{24}.NH_3$.

That is, the minimum molecular weight assignable to analcite is represented by four times its empirical formula. Other interpretations of the evidence are possible, but this appears to be the simplest. The water of analcite, as Friedel has shown, must be regarded as water only, not as hydroxyl, for it can be expelled by heat without destruction of the crystalline nucleus, the anhydrous salt, and is taken up again from moist air. But whatever its mode of union may be, the amount of water corresponds to the simple molecular ratio which is indicated in the formula of the species. One molecule of analcite holds a certain definite number of water molecules, and Friedel's observations are not incompatible with the idea that these are retained with varying degrees of tenacity. This idea is suggested by the various series of fractionation experiments which have been made from time to time by independent workers, even though the data are not by any means concordant. Thus, Lepierre (*Bull. Soc. Chim. de Paris*, 1896, Series 3, vol. xv., p. 561) found that half the water of analcite was driven off at or below 300°, the other half above 440°. In our own experiments three-fourths was expelled at 300°, the remaining fourth being held up to a much higher but undetermined temperature. In both series the water fractions are representable by fourths, but Friedel's experiments (*Bull. Soc. Min.*, 1896, vol. xix., p. 363) indicate a continuity of loss in weight of quite a dissimilar order. Friedel holds that all of the water fractionations heretofore made upon analcite are fallacious, and that no definite fractions can be identified, a conclusion strongly supported by his own data, even though the proof is not absolutely positive. The most that can be said is that the weight of evidence so far is in favour of Friedel's contention, but that additional investigation is necessary in order to reconcile all discrepancies. The full significance of the water in analcite remains unknown.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, January 16th, 1902.

Prof. Emerson Reynolds, Sc.D., V.P.R.S., President, in the Chair.

(Concluded from p. 56).

*2. "*The Constitution of Hydrocyanic, Cyanic, and Cyanuric Acids.*" By F. D. Chattaway and J. M. Wadmore.

A study of the behaviour of cyanogen chloride, bromide, and iodide shows that they possess the typical and characteristic properties of compounds in which halogen is attached to nitrogen.

They react, for example, quantitatively with solutions of hydriodic acid, sulphurous acid, and hydrogen sulphide, hydrocyanic acid being in each case reformed, whilst iodine, sulphuric acid, and sulphur respectively are produced.

They must, consequently, be regarded as imino-derivatives, and be represented by the constitutional formulæ,—

C:N·Cl C:N·Br C:N·I.

The ease with which the cyanogen halogen compounds can be formed from hydrocyanic acid and its salts, and again transformed into them, makes it in the highest degree probable that these also have the imino-constitution, and must be represented by formulæ such as—

C:N·H C:N·K C:N·Ag.

Since the cyanides and cyanogen halogen compounds have the above structure, their relations with cyanic acid

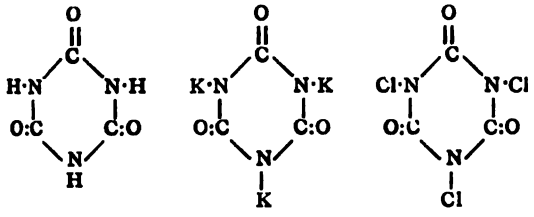
and the cyanates point to the probability of the latter also having the *iso*-constitution :—

O:C:N·H O:C:N·K O:C:N·Ag.

Cyanuric acid also yields a derivative in which all its hydrogen is replaced by chlorine, and which has the composition $C_3O_3N_3Cl_3$. It is formed by the action of chlorine on a solution of potassium cyanurate. It is a white powder, insoluble or soluble only with decomposition in most ordinary solvents, which under the microscope is seen to consist of short prisms, m. p. 245°.

Its behaviour shows that the whole of its chlorine is attached to nitrogen. It liberates chlorine when treated with hydrochloric acid, iodine with hydriodic acid, and oxidises sulphurous to sulphuric acid; it reacts explosively with a strong solution of ammonia, nitrogen being evolved, and also with a solution of hydrogen sulphide, setting free sulphur; cyanuric acid is in each case reformed.

Since cyanurates are readily and completely converted into this trichlorimino-derivative, and the latter in many reactions equally readily and completely again into cyanuric acid, the conclusion is justified that the latter and its derivatives are imino- and not hydroxy-compounds, and assuming the ring structure, they must therefore be represented by the formulæ :—



A similar study of the behaviour of cyanuric chloride and bromide shows that in them the halogen is attached to carbon and not to nitrogen.

Discussion.

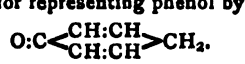
Professor Dobbie said that in a paper (Hartley, Dobbie, and Lauder, *Trans.*, 1901, lxxix., 848) on the absorption spectra of cyanogen compounds, it had been pointed out that if cyanuric acid possessed the formula usually assigned to it, it differed from all substances hitherto examined in which carbon and nitrogen atoms were united by alternate double and single bonds in showing no selective absorption. This was the case, however, not only with the derivatives of cyanuric, but also of those of *iso*cyanuric acid, so that if cyanuric acid possessed the imino-structure attributed to it by the authors, there still remained the difficulty of satisfactorily representing the constitution of the *iso*-derivatives. All that spectroscopic examination proves is that it is extremely unlikely that any of these compounds possess a structure analogous to that of pyridine or dimethylpyrazine.

Mr. C. E. Groves said that the very interesting substance, $C_3O_3N_3Cl_3$, to which the authors had given the name of trichloraminocyanuric acid, seemed to have rather the properties of a hypochlorite. Several ring compounds were known somewhat analogous to it in properties, of which the constitutions were accurately known in so far that the chlorine was certainly not attached to nitrogen; such, for instance, was the compound obtained by chlorinating trichloro-orcinol, $C_6Cl_3Me(OH)_2$, which might be represented by the hypochlorite formula, $C_6Cl_3Me(OCl)_2$. If this view were adopted, the formula of the new substance might be represented as $C_3N_3(OCl)_3$, in harmony with that usually assigned to cyanuric acid, $C_3N_3(OH)_3$.

Dr. Lapworth thought that, even if the halogen derivatives referred to had the constitutions which it was proposed to assign to them, it did not follow that the acids from which they are obtained had the same structure. The conclusions drawn were no doubt correct, but this had been decided by physical methods. Reasoning based on the assumption that a substituting atom or group takes up the position originally occupied by the atom displaced could only be accepted with the utmost caution when the molecule is of a labile type.

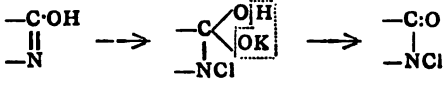
Thus, in the replacement of the metal in ethyl sodioacetoacetate by alkyls, the new group becomes attached to the carbon atom, but it is not generally conceded that the metal in the original compound occupied that position. Again, by the action of bromine water on phenol, a tetrabromo-substitution product having the structure—

is obtained (*Ber.*, 1900, xxxiii., 674), but this is not a sufficient reason for representing phenol by the formula—



In such cases the reactions are open to several interpretations, and in the present case the substitution experiments may be explained equally well by using the alternative formulæ for the acids.

Dr. Forster pointed out that the conversion of cyanuric acid into the chloro-derivative by the action of chlorine on a solution of the acid in potassium hydroxide is an argument which supports the hydroxylic representation of cyanuric acid quite as strongly as the iminic, the change represented by the formula,—



leading easily from hydroxylic cyanuric acid to the nitrogen chloride derivative.

Dr. Travers remarked that though the authors had referred to the work of Hartley and others as supporting their theory, the researches of these same investigators on isatin and its methyl derivatives pointed to the danger of accepting such conclusions. Since *ketonic* isatin gives rise, by treating its salts with methyl iodide, to *enolic* methyl isatin, obviously no conclusion could be drawn from such a reaction, which they explain on the basis of the ionic theory.

Professor Dunstan was of opinion that in general the tautomerism of these cyanogen derivatives renders it impossible to assign to any one of them a formula which will satisfactorily account for all its reactions. The authors had obtained a very interesting chlorine derivative of cyanuric acid, but its behaviour did not appear to be inconsistent with its being regarded as containing chlorine in the place of the hydrogen of three hydroxyl groups. He considered improbable the authors' explanation of its decomposition by hydrochloric acid giving free chlorine.

Dr. Chattaway, in reply, said that their work neither proved nor disproved the cyclic constitution of cyanuric acid; it only showed that, whatever the formula, the whole of the hydrogen was attached to nitrogen, and therefore the hydroxylic formula was not possible.

The stability of the chlorine derivative of cyanuric acid, and the analogy of its reactions with those of substances such as nitrogen iodide and cyanogen iodide, showed that it could not contain —O—Cl groups.

*3 "*A Modification of Zeisel's Method for the Estimation of Methoxyl Groups.*" By J. T. Hewitt and T. S. Moore.

The authors described a modification of Zeisel's well-known method in which the condenser supplied with water at 40° is replaced by a fractionating column; this is so efficient that the potash bulbs containing water with red phosphorus in suspension may be dispensed with. The time taken in fitting up the apparatus and in carrying out the operation is much less than in Zeisel's original method. Experimental data were given showing the accuracy attainable.

*4. "*A New Colour Reaction of Hydroxylamine.*" By W. C. Ball, B.A.

During the course of some experiments on the action of hydroxylamine salts on nickel sulphide, the author obtained an intense purple colouration, which proved eventually to be independent of the presence of nickel or any other metal.

The production of this colour is a very delicate test for hydroxylamine, as solutions so dilute as no longer to reduce Fehling's solution perceptibly, either in the cold or on boiling, easily respond to this test. The best method of applying the reaction is to boil the hydroxylamine solution with one or two drops of yellow ammonium sulphide solution until a precipitate of sulphur is produced; two or three cubic centimetres of 0·880 ammonia solution are then added, and finally an equal volume of strong pure alcohol.

The purple colour thus developed exhibits a characteristic absorption spectrum, consisting of a wide band covering the yellow, orange, and part of the green. In stronger solutions, the violet and blue are absorbed, and in very strong solutions the green rays also, so that the only light transmitted is a band in the red.

With a dilution of 1 part of hydroxylamine in 2000 of water the colouration is intense, and the band very distinct; with a dilution of 1 part in 300,000 of water, the colouration is still strong, and the band perceptible in 3 centimetres thickness of the solution. By means of this test hydroxylamine may be detected when the dilution does not exceed 1 part in 500 000 of water.

The production of the colour is well adapted for showing the formation of hydroxylamine by the action of metals, for example, tin, on nitric acid. It also serves as a very good means of distinguishing hydroxylamine from hydrazine, as the latter base does not produce the colour. The coloured substance is also produced, and can be obtained in the solid form, by the action of dry ammonia on an alcoholic solution of sulphur monobromide, or an acetone solution of nitrogen sulphide in presence of a little hydrogen sulphide.

The colouring matter seems to be the same as that observed by Fordos and Gélis when nitrogen sulphide containing sulphur is treated with alcoholic potash.

The absorption spectrum of the compound appears to be identical with that of Moissan's sulphammonium, obtained by the action of liquid ammonia on sulphur.

The author is endeavouring to obtain the substance pure enough for analysis.

*5. "*On the Sensitiveness of a Thermoregulator.*" By A. W. C. Menzies.

The following details have been recorded, as it is not generally known within what small limits of temperature a bath may be kept constant, using gas heating and the ordinary form of regulator. The glass "horse-shoe" reservoir was of the usual form, with a capacity of 390 c.c. and an outer surface of about 650 sq. c.m.; to this reservoir the ordinary U-form regulating tube which contains mercury, was fused on, and the glass connections so bent that all the tubes containing enclosed liquid were under the water of the bath. As toluene was used for the expanding liquid, the stop-cock that closes one limb of the U-tube was lubricated with syrupy phosphoric acid. The bath held 14 litres and was of enamelled iron, with no jacket. The stirrer was driven by a small electromotor, and care was taken that a portion of the vibration of the motor was communicated to the regulator, thus aiding the free movement of the mercury in response to the pressure of the toluene. The gas supply to the by-pass was adjusted by a screw-clip in such a way that the by-pass flame alone was nearly sufficient to maintain the temperature. Using a tube of 3·1 m.m. bore for the outer limb of the regulator, the temperature of the bath can be kept constant at 18° within a total range of 0·008°, that is, 0·004° on each side of the middle point. After twenty-four days, using unfiltered coal-gas, the actual temperature was the same as at starting, and the closeness of regulation was found still within the above limits. If narrower tubes be used, it is well to dry and filter the gas. The inlet tube that leads the gas down to the surface of the mercury is cut off square, and should be so wide that on trial the mercury rises higher in it than in the space between it and the regulator tube. A bore of 1·9 m.m. for the outer limb of the regulator gave a temperature variation in the bath of not more (though probably less) than 0·0025 (total range). The difference of temperature between bath and room was 6—7°; if the flame were removed, the bath was found to cool 0·1° in 4·5 minutes. With this regulator, a well-tapped calorimetric thermometer by Golaz, divided into fiftieths of a degree, remained apparently stationary to an estimated tenth-division, as also did a "Beckmann" thermometer graduated directly to hundredths. The limits actually registered by two large special alcohol thermometers (one with a bulb of extremely thin glass 180 m.m. long and 5 m.m. in diameter) were less than those stated above, which are given after making due allowance for the sluggishness of action of such thermometers.

6. "*Myricetin.*" Part II. By A. G. Perkin.

Myricetin (*Trans.*, 1896, 1287), air-dried, has the formula $C_{15}H_{10}O_8,H_2O$, is anhydrous at 160°, and melts about 357°. The compound previously obtained by the action of bromine is *tetrabrom-myricetin*, $C_{15}H_6Br_4O_8$, and is converted hy hydriodic acid into myricetin. *Myricetin-pentamethyl ether*, $C_{15}H_5O_3(OCH_3)_5$, m. p. 138—139°, forms a *monoacetyl* derivative, $C_{15}H_4O_3(OCH_3)_5(C_2H_3O)$, colourless needles, m. p. 167—170°, and on decomposition gives gallic acid trimethyl ether and phloroglucinol monomethyl ether, the latter identified by its diazobenzene derivative (m. p. 250—252°). *Myricetinhexaethyl ether*, $C_{15}H_4O_2(OEt)_6$, almost colourless needles, m. p. 149—151°, yields gallic acid triethyl ether and a phenol, probably phloroglucinol diethyl ether (azobenzene compound, yellow needles, m. p. 163—165°). Acetylmyricetin melts at 211—212° and not at 204—206° as previously stated.

Myricetrin, air-dried, $C_{21}H_{22}O_{13},H_2O$; at 160°, $C_{21}H_{22}O_{13}$, pale yellow leaflets, m. p. 199—200°; a new glucoside, occurs in the bark of the *Myrica nagi*, and is decomposed by acid into myricetin and rhamnose, $C_{21}H_{22}O_{13}+H_2O=C_{15}H_{10}O_8+C_6H_{14}O_6$. It closely resembles quercitrin.

7. "*The Colouring matters of Green Ebony.*" By A. G. Perkin and S. H. C. Briggs.

Green ebony is a yellow dyewood, probably *Excoecaria glandulosa* or *Jacaranda ovalifolia*, until recently employed in this country. It contains two crystalline colouring matters, excoecarin and jacarandin, in minute quantity. *Excoecarin*, $C_{13}H_{12}O_5$, yellow needles, m. p. 219—221°, a weak, substantive dyestuff with animal fibres, forms a *tribenzoyl* compound, $C_{13}H_9O_5(C_7H_5O)_3$, colourless needles, m. p. 168—171°, and a *dimethyl* ether, m. p. 117—119°, yellow needles, giving fluorescent solutions. On fusion with alkali, it yields *hydrotoluquinone* ($CH_3:O:O=1:2:5$), and *hydroquinone carboxylic acid*, the latter apparently derived from the former. Bromine in presence of alcoholic potassium acetate forms *excoecarone*, $C_{13}H_{10}O_5$, copper coloured needles, m. p. about 250°, which on reduction is re-converted into excoecarin. Alcoholic quinone solution gives a similar compound ($C_{19}H_{14}O_7$?), green leaflets, m. p. 190°, which is possibly a quinhydrone derivative. *Jacarandin*, $C_{14}H_{12}O_5$, yellow plates, m. p. 243—245°, resembles luteolin in dyeing property, and gives a *diacetyl*, $C_{14}H_{10}O_5(C_2H_3O)_2$, yellow needles, m. p. 192—194°, and a *dibenzoyl* derivative, $C_{14}H_{10}O_5(C_7H_5O)_2$, yellow prismatic needles, m. p. 167—169°. With alcoholic potassium acetate, the *salt*, $C_{28}H_{23}O_{10}K$, yellow needles, results, and on fusion with alkali, an acid, apparently derived from catechol, is formed. The wood contains also two orange-coloured resins, one of which is a yellow dyestuff, but the other is devoid of tinctorial property.

8. "*The Action of Methylene Iodide on Aryl- and Naphthyl-amines: Diarylmethylenediamines, Acridines, and Naphthacridines.*" By A. Senier and W. Goodwin.

The compounds obtained by the interaction of methylene iodide and arylamines (*Trans.*, 1901, lxxii., 254) were believed to be arylmethylenediamines. The study of these compounds now proves that in the case of the reaction with aniline, the toluidines, and xylidine, such diamines are formed. With ψ-cumidine, however, the well-defined, beautiful, yellow, crystalline compound which results is shown to be a condensation product. Its analysis, vapour density, the fluorescence of its solutions, and the analogy of its formation to the anthracene synthesis of Friedel and Crafts (*Ann. Phys.*, 1887, [vi.], xi., 263), and the acridine synthesis of Bernthsen and Bender (*Ber.*, 1883, xvi., 1802), indicate it to be an acridine:—hexamethylacridine. This view is confirmed by the exactly parallel behaviour of both α- and β-naphthylamine with methylene iodide, giving rise to similar compounds, and the identity of the β-compound with the β-naphthacridine already known. This naphthacridine was discovered by Reed, using a different reaction (*Journ. Prak. Chem.*, 1886, [ii.], xxxiv., 160; 1887, xxxv., 298), and has led to interesting results in the hands of Morgan (*Trans.*, 1898, lxxiii., 536).

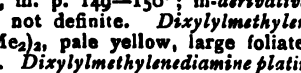
The further study of the properties and constitution of hexamethylacridine and α-naphthacridine is being proceeded with, and is reserved for a future communication.

The following compounds were described:—*Diphenylmethylenediamine*, $CH_2(NHPh)_2$, dark yellow powder, m. p. 65—67°. *Diphenylmethylenediamine platinichloride*, $(C_{13}H_{14}N_2)H_2PtCl_6$, olive-green powder. *Dicarbanilidodiphenylmethylenediamine*, $CH_2(NPh{\cdot}CO{\cdot}NHPh)$. *Ditolylmethylenediamines*, $CH_2(NHC_6H_4Me)_2$; *o-derivative*, colourless crystals, m. p. 156—157°; *p-derivative*, pale yellow crystals, m. p. 149—150°; *m-derivative*, yellow, amorphous but not definite. *Dixylylmethylenediamine*, $CH_2(NHC_6H_3Me_2)_2$, pale yellow, large foliate crystals, m. p. 127—128°. *Dixylylmethylenediamine platinichloride*, $(C_{17}H_{22}N_2)H_2PtCl_6$, golden-yellow powder. *Dicarbanilidocarbanilidodixylylmethylenediamine*,—

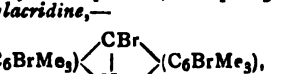
$$CH_2[N(C_6H_3Me_2){\cdot}CO{\cdot}NPh{\cdot}CO{\cdot}NHPh]_2,$$

yellowish-white substance resembling wax, yielding colourless needles when re-crystallised, m. p. 203°.

Hexamethylacridine,—

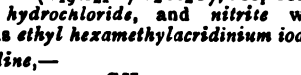


fine yellow needles, m. p. 221—222°, sublimes readily, solutions fluorescent. *Hexamethylacridine picrate*, $(C_{19}H_{21}N)C_6H_2(NO_2)_3OH$, fine, brown, plume-like crystals, m. p. 200—203°. *Dinitrohexamethylacridine*, $C_{19}H_{19}(NO_2)_2N$, yellow powder, m. p. 85—87°. *Tribromohexamethylacridine*,—

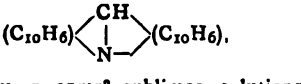


red. *Hexamethylacridine nitrate*, $(C_{19}H_{21}N)HNO_3$, bright scarlet needles, m. p. 163—164° with decomposition. *Hexamethylacridine platinichloride*, $(C_{19}H_{21}N)_2H_2PtCl_6$, scarlet needles. *Hexamethylacridine aurichloride*, $(C_{19}H_{21}N)HAuCl_4$, yellow powder. *Hexamethylacridine mercurichloride*, $(C_{19}H_{21}N)_2HgCl_2$, dark red, glistening needles with remarkable yellow lustre. *Hexamethylacridine dichromate* $(C_{19}H_{21}N)H_2Cr_2O_7$, deep red crystals. The *sulphate*, *hydrochloride*, and *nitrite* were also described, as was *ethyl hexamethylacridinium iodide*.

α-Naphthacridine,—



pale-yellow crystals, m. p. 173°, sublimes, solutions fluorescent. *Mononitro-α-naphthacridine*, $C_{21}H_{12}(NO_2)N$, m. p. 105—107°. *α-Naphthacridine platinichloride*, $(C_{21}H_{13}N)_2H_2PtCl_6{,}2H_2O$. *α-Naphthacridine picrate*, $(C_{21}H_{13}N)C_6H_2(NO_2)_3OH$, scarlet needles, m. p. 176—178°. *β-Naphthacridine*,—



yellow needles, m. p. 215·5°, sublimes, solutions fluorescent, identical with β-naphthacridine of Reed. *β-Naphthacridine platinichloride*, $(C_{21}H_3N)_2H_2PtCl_6(H_2O)_2$, yellow powder.

9. "*The Polmerisation of Cyanic Acid: Cyanuric Acid and Cyamelide.*" By A. Senier and T. Walsh.

In the polmerisation of liquid cyanic acid, cyamelide is not the only product. The two isomerides, cyamelide and cyanuric acid, are formed. These may be separated by treatment with water. Pure cyamelide so obtained is soluble in water to the extent of 0·01 per cent at 15°. The solubility of cyanuric acid at 15° is from 0·145 to 0·16 per cent. Phosphorus pentachloride has no action on cyamelide.

NOTICES OF BOOKS.

The Life of Pasteur. By René Vallery-Radot. Translated from the French by Mrs. L. L. Devonshire. In Two Volumes. London: Archibald Constable and Co., Limited. 1902.

In the "Life of Pasteur," by René Vallery-Radot, we have a work of more than usual interest, written by one in the position of closest intimacy. The title of the work has been fully substiantiated; we have not merely a recapitulation of the labours of this truly great man, but we are brought into intimate acquaintance with the man himself. To English readers the thought might occur that the author with pardonable enthusiasm had introduced matters almost outside the bounds of the narrative, but the effect is only to enhance the interest in the work by giving an insight into French thoughts and customs that otherwise would only be realised by those who have had the privilege of experience in French scientific circles; in this way the reader is brought into close touch with such names as Biot, Ste. Claire Deville, J. B. Dumas, Regnault, and many others; even the chapter dealing with the war and the siege cannot be looked upon altogether as a digression, for it is legitimately introduced to bring out the extraordinary combination of science and patriotism found in this remarkable man. In studying the work the personality of Pasteur stands prominently before everything. Born in very humble circumstances, his father, a retired sergeant-major (though decorated with the ribbon of the Legion of Honour), carrying on the business of a tanner in the little town of Arebois, Louis Pasteur had to make his position in the world; fortunately his desire for a scientific career was encouraged by his father, but hard work, and plenty of it, was the road by which he had to travel, and the difficulties in the way only helped to stimulate a mind set upon success. There is a sentence in a letter from Pasteur, then a youth of eighteen holding the position of preparation master in the Royal College, Besançon, which is worth quoting:—"Three things, *will*, *work*, *success*, fill human existence; *will* opens the door, *work* passes the door, and at the end *success* comes to crown our efforts." Looking back fifty years afterwards he might well have summed up the whole of his labours with those three words.

As son, husband, and father, Pasteur seems to have had the share of trouble that falls to the lot of most mortals, but nothing could damp his insatiable desire to win

nature's secrets; the more difficult and dangerous the work the greater the delight in working, and yet he could never be charged with selfishness, but was ever ready to help and courteous to listen to both inquirers and critics; his modesty, depth of feeling, and religious convictions are related by the author with a simplicity that testifies of their genuineness; his oration at the funeral of his friend Ste. Claire Deville, and the kindness of heart that could keep him for a whole day at the bedside of a poor child who had been bitten by a mad dog, but not brought to him until too late for his treatment to take effect, and his tearful words to the father and mother, "and I did so wish I could have saved your little one," completely refute those fanatics who would brand him as a heartless vivisectionist.

The reality of his patriotism is shown, when in his young days he could place the whole of his savings of one hundred and fifty francs on the "autel de la patrie," and when later in life, in those dark days following the war, he refused a professor's chair at Pisa, with very great personal advantages, with "I should feel that I deserved a deserter's penalty if I sought away from my country in distress a material situation better than it can offer me." His feelings against his country's conquerors at that time were bitter indeed, and we find him writing to his pupil and friend M. Raulin, "every one of my future works will bear on its title-page 'Hatred to Prussia. Revenge! Revenge!'"—feelings that were softened but not altered, when, twenty years afterwards, he could courteously refuse the badge of the "Order of Merit" from the German Academy of Sciences.

Of Pasteur's long list of discoveries the first, and the one that brought him prominently before the scientific world, was his work on the optical properties and origin of tartaric and para-tartaric or racemic acid, and there is introduced a happy touch in the life of Biot, then seventy-four years of age. Biot having by careful personal verification satisfied himself of the genuineness of Pasteur's discovery, took the young man by the arm with the words, "My dear boy, I have loved science so much during my life that this touches my heart." But the greatest works of Pasteur began with his study of fermentations, and from that moment until the end of his life his labours are one unbroken record of triumphs; the study of the "infinitely small" brought results beyond the range of any human expectations; the settlement of the vexed question of spontaneous generation, a question beset with such difficulties that even his friend and patron Biot tried hard to stop him. "You will never find your way out," cried Biot; "I shall try," said Pasteur, and the result is a matter of history. The prevention of silkworm disease, treatment of splenic fever, puerperal fever, chicken cholera, the application of his discoveries to the antiseptic treatment in surgery, applied first with such wonderful success by Lister in England, and now universally in every hospital in the world; the investigation of rabies, with the treatment for hydrophobia and diphtheria, alone place Pasteur as one of the greatest benefactors of the human race; to him was granted the rare good fortune of living to reap in some measure the fruit of his labours. His hearty reception in the Academy of Sciences, gift of the Legion of Honour, substantial testimonial of gratitude from his countrymen, honours from every country in the world, must have helped much to lighten the bodily infirmity of his later years; but what must have appealed to the heart of the man more than any of these things was the thought of a "little Joseph Meister" saved from a death too horrible to contemplate, the life of the brave shepherd boy J. B. Jupille, women in lying-in hospitals, patients operated upon in hospitals and upon the battle-field, thousands of human beings whose existence had been saved by his methods—rewards of a far higher order than any social distinctions or recognition.

Science, said Pasteur, has been the "ruling passion in my life"; never before has the pursuit of science been so directly connected with the alleviation of human suffering.

Elementary Experimental Chemistry, Inorganic. Completely illustrated with Full-page Engravings of all the Apparatus and Chemicals used in Experiments. For Students in High Schools and Junior Classes in Colleges, and Private Learners. By W. F. Watson, A.M., Professor of Chemistry and Biology in Furman University, South Carolina. New York: A. S. Barnes and Co. 1901.

The author has succeeded in his endeavour to "present the fundamental principles of elementary chemistry in a brief, clear, and logical manner," and throughout the work no pains have been spared by the author to make his meaning clear. A novel feature in the book is the introduction of each subject by a full-page illustration from a photograph of the apparatus and materials to be used in the experiments about to be described; this is quite a new departure, and is the outcome of the desire to save labour both to the instructor who is using the work and to the student. We must confess to the feeling that both by this device are in a way reduced to the level of automata, and it does *not* tend to "develop the observing and reasoning faculties" of either. Some of these full-page engravings, although doubtless possessing artistic merit, have but little connection with the subject; for instance, "The Metals" is introduced by a picture of some shelves, with a hammer, some photographic dishes, a very fine rack of test-tubes, and a number of bottles apparently partly full of something, but with labels unreadable. However, as we have already pointed out, the subject matter is simple, clear, and to the point, and this is of the most importance. We are glad to see that in the list of elements didymium has been eliminated, and neo- and praseo-dymium introduced. There is an appendix containing much useful information and tables, including an extremely useful one of the solubilities of metallic salts. The book is well indexed, and has a table of contents.

The Laboratory Companion to Fats and Oils Industries. By Dr. J. Lewkowitsch, M.A., F.I.C. London: Macmillan and Co., Limited. New York: The Macmillan Company. 1901.

This book consists almost exclusively of tables. For explanations and description of methods the reader is referred to the author's "Chemical Analysis of Oils, Fats, and Waxes, and of the Commercial Products Derived Therefrom" (Second Edition, Macmillan and Co., 1898). A few pages of subject matter are added explanatory of the system of testing oils and fats from results of more recent date than the work on chemical analysis. Many of the tables and values extracted from the author's laboratory note-books are now published for the first time.

This collection of tables and data will undoubtedly be of considerable value to the works chemist. There is a table of contents and a very complete index.

CORRESPONDENCE.

THE INSTITUTE OF CHEMISTRY.

To the Editor of the Chemical News.

Sir,—Dr. A. Wynter Blyth's fear that the Council of the above body will "cease to be respected" has, I am afraid, good justification, and on other grounds than that mentioned in his letter to the Chemical News of January 24th.

Judging from the frantic efforts of the Council to draw the Associates within the fold of the Fellowship, they must have grave fears that something like 80 per cent of the Associates do not think it worth while to proceed to the higher qualification. If this is so it is surely an in

dication that something is wrong, and it is high time the members took more interest in the doings of the Council, and insisted upon the object of the incorporation being carried out. If the Institute is to remain merely an examining body why accumulate funds? What has it done to raise the status of the chemical profession? I am aware that some half-hearted effort was made to secure legal recognition, but still the fact remains that it has accomplished nothing but a satisfactory balance-sheet and a club-house for the London members.

Surely with the ample funds and income at its disposal it can now do something towards the object of its existence without extorting further guineas from not too well off Associates.

Let the Institute do something to justify confidence in it, and the Associates will be eager enough to proceed to the Fellowship; but to charge four or five guineas for changing the letter A to F (for that is all it amounts to), is a trifle arbitrary even if it is not illegal.

"Hope deferred maketh the heart sick." The Institute has now been in existence for twenty-five years, and during the past seventeen years with a Royal Charter. Yet it has no journal or other organ; it has no provincial library, it gives no certificates of competency, it has done nothing to advance the well-being of the members, legally, professionally, socially, or benevolently; truly a more miserable record of inactivity and failure it would be hard to find.—I am, &c.,

AN ASSOCIATE.

Liverpool, February 5, 1902.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxiv., No. 2, January 13, 1902.

Preparation and Properties of Sodium Hydride.—Henri Moissan.—Sodium, like potassium, gives a crystalline hydride of formula NaH, which possesses most energetic reducing properties and is soluble in the alkaline metals. This substance is prepared by slowly raising the temperature of sodium in an atmosphere of hydrogen gas. The hydride thus prepared is in the form of transparent crystals, which are decomposed by slight traces of water. The existence of this new compound shows that the alkaline metals, as well as the metals of the alkaline earths, can be made to unite with hydrogen directly, to give perfectly crystalline and definite compounds, decomposable by water, and more or less dissociable by simple elevation of temperature. These form a new series of curious compounds possessing important properties.

Radio-active Bodies.—P. Curie and Mdme. S. Curie.—During their extensive researches on the subject of radio-active bodies, the authors admit that the radio-activity is an atomic property of the body. Each atom of such a body acts as a constant source of energy. From this hypothesis very varied consequences can be deduced, which can be confirmed by experiment without the necessity of definitely deciding from whence this energy is obtained. The experience of several years shows that for uranium, radium, thorium, and probably also for actinium, the radio-activity is rigorously the same whenever the body is brought to the same chemical and physical state, and this activity does not vary with time.

Preparation and Properties of Strontium Hydride.—Henri Gautier.—Strontium hydride, whose existence has already been announced by the author, is found to be a reducing agent. From its composition, and from a study of its properties, it apparently is very similar to calcium hydride, CaH_2.

Chemical Equilibrium of Iron-carbon System.—Georges Charpy and Louis Grenet.—A study of various specimens of iron-carbon metal leads the authors to the following conclusions:—(1) The separation of graphite proceeds to a temperature considerably lower than that at which the retention of silicon is the greatest. (2) The separation of graphite, once begun, continues to temperatures below those at which the reaction could be started. (3) At a constant temperature the separation of graphite is effected progressively with a rapidity which decreases as the temperature is lowered and as the retention of silicon becomes more feeble. (4) The dissolved graphite which is present at equilibrium point appears only to depend very slightly on the quantity of silicon present. (5) The dissolved graphite corresponding to equilibrium point increases when the temperature is lowered, and at low temperatures the equilibrium appears to correspond to the absence of combined carbon.

Action of Mixed Organo-Magnesium Compounds on Trioxymethylene. Syntheses of Primary Alcohols.—V. Grignard and L. Tissier.—Whilst free aldehydes react with great energy on organo-magnesium compounds, trioxymethylene only depolymerises slowly in contact with these substances; the reaction is scarcely perceptible and prolonged heating is necessary. Using special precautions the authors obtain excellent yields, and prove that in the aromatic series, as well as in the fatty series, a certain number of syntheses of primary alcohols are possible, which fact testifies to the generality and practical value of the use of organo-metallic compounds.

Preparation and Properties of Imido-dithio-carbonic Ethers.—Marcel Delépine.—In a previous research the author described the methods of formation of the imido-dithio-carbonic ethers $RN=C(SR')_2$, and justified their constitution by their synthesis, their oxidation, their reduction, and their decomposition. He now describes a more definite preparation, and examines the properties of this compound.

The Inversion of Saccharose.—P. Petit.—The heat evolved by the inversion of saccharose can be deduced from the heats of combustion and solution. The author endeavours, however, to determine this quantity directly for dissolved saccharose by using sulphuric acid. As a mean of a number of experiments he finds that the heat evolved by the inversion of a gramme-molecule of saccharose dissolved in 140 molecules of water is 2·639 cal. at 58·5°, and 2·675 cal. at 63°. The author proposes to repeat these experiments with sucrose, and to apply the same method to an examination of the products of saccharification of amidon by amylase.

The Solubility of Bi calcic Phosphate in Pure Water.—A. Rindell.—The constants of solubility of different phosphates of lime being very incomplete and even contradictory, the author undertakes an investigation of this subject with modern methods. He uses a very pure salt, finely pulverised, and having the following composition:—CaO, 32·70; P_2O_5, 41·30; H_2O, 26·20 per cent. The numbers obtained show clearly (1) that the concentration of the solution increases with the time and the mass of the salt in contact with a given volume of water; (2) the quantity R at first differing but slightly from unity increases with the concentration.

The Methods of Volumetric Estimation by Stannous Chloride of Copper, Iron, Antimony, Powdered Zinc, Sulphur in Sulphides, Glucose, and Sugar.—F. Weil.—The author found some years past that instead of effecting the above titrations at boiling-point, they could be equally well performed at ordinary temperatures. It is necessary to introduce 10 c.c. of the solution to be titrated to 30 c.c. of hydrochloric acid and some fragments of white marble. The carbon dioxide which is evolved prevents the oxidation of the cuprous chloride produced

during the reaction. The advantage gained by working at ordinary temperatures is the absence of hydrochloric vapours, which are evolved in great quantity when the solutions are treated at boiling-point.

Mechanism of Synthesis of Leucine Isomer.—A. Etard and A. Vila.—Leucine which is derived by the synthesis of amylic alcohol is different from biological leucine. It is formed as a result of a series of reactions which can be controlled. The leucine of synthesis possesses two asymmetric carbons, and when it is decomposed certain actions take place on the cellules which ordinarily do not occur.

MISCELLANEOUS.

Institute of Chemistry Examinations.—Pass Lists (January, 1902).—*Intermediate Examination*: G. J. Alderton; K. B. Benham; J. L. Garle; H. W. Hill; J. Johnston; A. W. Knapp, B.Sc. (Birm.); S. G. Paine; W. B. Parker; J. B. Pursglove; J. C. Sheldon; H. A. Tempany; and R. Tyler. *Final A.I.C. Examination* (*Branch* "A," *Mineral Chemistry*): A. G. Armstrong; W. W. Lumsden; A. B. Shepherd, B.Sc. (Vict.); and W. F. Sutherst, Ph.D. (Geneva). *Branch* "D" (*Organic Chemistry*): G. Clarke, jun.; E. D. M. Neuman, B.A. (Oxon.), Ph.D (Göttingen); H. A. D. Neville, B.Sc. (Lond.); G. M. Norman, A.R.C.Sc., B.Sc. (Lond.); W. H. Nuttall; W. H. Peters, and V. W. Theobalds. *Branch* "E" (*Analysis of Food and Drugs, including an Examination in Therapeutics, Pharmacology, and Microscopy*): F. W. F. Arnaud; J. Evans; J. E. Jenkins; W. Partridge; S. O. Richmond; and E. C. Spurge.

Prize for Industrial Hygiene.—The Council of the Society of Arts are prepared to award, under the terms of the Benjamin Shaw Trust, a Gold Medal, or a prize of £20. The Medal, under the conditions laid down by the testator, is to be given "for any discovery, invention, or newly-devised method for obviating or materially diminishing any risk to life, limb, or health, incidental to any industrial occupation, and not previously capable of being so obviated or diminished by any known and practically available means." Intending competitors should send in descriptions of their inventions not later than May 1, 1902, to the Secretary of the Society of Arts, Adelphi, London, W.C. Such descriptions may be sent in under the inventor's name, or under a motto, accompanied by a sealed envelope enclosing the name as preferred. The Judges will be appointed by the Council. The Council reserve the right of withholding the prize or of awarding a smaller prize or smaller prizes, if in the opinion of the Judges nothing deserving the full award is sent in.

MEETINGS FOR THE WEEK.

MONDAY, 10th.—Society of Arts, 8. (Cantor Lectures). "Personal Jewellery from Prehistoric Times," by Cyril Davenport.

TUESDAY, 11th.—Royal Institution, 3. "The Cell—its means of Offence and Defence—Immunity," by Allen Macfadyen, M.D., B.Sc.

WEDNESDAY, 12th.—Society of Arts, 8. "Industrial Redistribution and its connection with the Overcrowding Question," by W. L. Madgen.

THURSDAY, 13th.—Royal Institution, 3. "The Scot of the Eighteenth Century—II., In Kirk," by the Rev. John Watson, D.D. (Ian Maclaren).

FRIDAY, 14th.—Royal Institution, 9. "Magic Squares and other Problems on a Chess Board," by Major P. A. MacMahon, R.A., D.Sc., F.R.S.

—— Physical, 5. (Annual Meeting). Address by the President. Mr. T. H. Littlewood will exhibit an Atwood's Machine.

SATURDAY, 15th.—Royal Institution, 3. "Some Electrical Developments," by Lord Rayleigh, M.A., F.R.S., &c.

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PHYSICS..	R. A. LEHFELDT, D.Sc.
CHEMISTRY	J. T. HEWITT, D.Sc.
ENGINEERING (Mech.).. ..	D. A. LOW, M.I.M.E.
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Applications, on forms which may be obtained at my office, stating age, experience, and qualifications, and accompanied by not more than three testimonials, to be sent to me, endorsed "Assistant," not later than 4 o'clock on MONDAY, FEBRUARY 17th, 1902.

Canvassing will disqualify.

J. A. JOHNSON,
Town Clerk.

Southwark Town Hall,
Walworth Road, S.E., February 5, 1902.

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Chemical Industry Journal, complete, 1882-1900, with Index. 20 vols., half roan. £24.

Chemical News, complete, 1860-98. 78 vols., orig. cloth. £22 10s.

Chemical Society Journal. 1888-1900. 20 vols. half cloth, remainder in parts. £10 17s. 6d.

Jahresbericht uber die fortschritte der Chemie, 1859-71, and 1874-83. Register, 1867-76. 24 vols. £15 15s.

Chemistry Applied to Manufactures and Art. 8 vols., 1880. 25s.

Spon's Encyclopædia of the Industrial Arts. 2 vols., 1882, half morocco. 55s.

Dumas' Traite de Chimie Applique aux Arts, with Atlas. 9 vols. 1828. 30s.

Textile Colourist, edited by O'Neil. 4 vols., 1876-7. 40s.

Pelouse et Fremy's Traite de Chimie Générale. 7 vols. 25s.

Year-Book of Pharmacy, 1870-96. £4 4s.

Pharmaceutical Journal and Transactions, 1841-99. 62 vols. £11.

Watts' Dictionary of Chemistry. 4 vols. £4 18s. 6d.

Thorpe's Dictionary of Applied Chemistry. 3 vols. £4 2s. 6d.

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THE SPECIFIC VOLUMES OF OXYGEN AND NITROGEN VAPOUR AT THE BOILING-POINT OF OXYGEN.*

By JAMES DEWAR, M.A., D.Sc., LL.D. F.R.S.,

In my paper on "The Boiling-point of Liquid Hydrogen Determined by Hydrogen and Helium Gas Thermometers" (*Roy. Soc. Proc.*, 1901, vol. lxviii.; CHEMICAL NEWS, vol. lxxxiii., p. 97) it was pointed out that a constant-volume gas-thermometer filled with oxygen gas, having a pressure at 0° C. of about 800 m.m., gave a very accurate value of the boiling-point of liquid oxygen. As it seemed advisable to confirm this result indirectly, an attempt was made to determine the vapour-density of oxygen at its boiling-point by direct weighing, the intention being, if the experimental results proved at all encouraging, to repeat the work on a larger scale and with greater precautions. As at present there is no likelihood of my being able to undertake the more accurate determinations, the results of the preliminary enquiry are presented to the Society. They give in any case, with considerable accuracy, the specific volumes which have never been directly determined.

In order to obviate any question of the buoyancy of the air, two flasks, A and B, of as nearly as possible the same air displacement, were counterpoised on an Oertling balance. The B flask remained permanently on one scale of the balance during all the weighings, while the A flask was weighed either exhausted or filled with oxygen (or nitrogen), under various circumstances according as the experiments required.

As the flask cooled in the liquid oxygen or air had to stand an internal pressure of from three to four atmospheres, it was considered expedient to select a spherical vessel of about 300 c.c. capacity, to which was sealed a narrow tube having a very carefully ground stopcock at its end. Preliminary experiments were made to determine the change of volume of the flask when subjected to internal pressure. The flask, filled with air at five atmospheres pressure, was left for twenty-four hours with the stopcock closed, without showing any leakage. To determine the effect of pressure on the capacity of the flask, it was filled with water under one atmosphere pressure, and again with water under three or four atmospheres pressure (which included the range of the observations) and the weights noted. From these the coefficient of expansion of the flask was found to be 0·000306 per atmosphere excess of internal over external pressure. The temperature coefficient of expansion of glass (cubical) was taken to be 0·000025, which for a variation of temperature of some 200° altered the capacity of the flask by about 1·5 c.c. The content of the flask up to a fixed mark on the neck below the stopcock was determined to be 315·973 c.c. at 17° C.; the content between the mark and the stopcock was determined both by measurement and by the weight of mercury it contained, and was found to be 0·127 c.c.

Before each experiment the A flask, filled with the gas under observation, was exhausted to a pressure of from 2 to 4 m.m. of mercury (which was afterwards involved as a correction in reducing the observations) and weighed, the weight a which had to be added to its scale pan to balance the B flask being noted. The A flask was then filled with carefully purified oxygen (or nitrogen), and the temperature of the flask and contents (still in communication with the gas reservoir) was lowered by immersing it up to the mark in liquid oxygen (or air) until the gas ceased passing into the flask, and the pressure was finally equalised to that of the atmosphere. This is really the most important part of the manipulation, as the accuracy mainly depends on giving sufficient time to the cooling, while at the same time taking care to avoid any excess of pressure that would necessarily lead to liquid condensation on the glass surface. During the rapid inrush of gas, it is advisable to keep the pressure well below that of the barometer at the time, and to finally adjust the pressure at the end of the absorption. The vessel containing the liquid oxygen had a cardboard cover which crossed the neck of the flask at the mark, thereby preventing the cooled vapour of the oxygen from freezing the stopcock. The remaining portion of the tube between the mark and the stopcock, namely, 0·127 c.c., was therefore approximately at the temperature of the room. When the temperature of the flask and its contents became stationary the stopcock was closed, the flask removed, and after heating up to the temperature of the room, weighed against the B flask and any further necessary counterpoise b. The weight of oxygen vapour in the flask at its boiling-point was thus equal to $a+b$, subject to the corrections which have been indicated.

To the five sources of error or correction indicated above, there remains to be added that due to additional buoyancy of the air during the weighing of the flask, when filled with oxygen at its boiling-point, for these weighings were made at the temperature of the room, which would cause a rise of pressure in the flask and therefore expansion of its volume. But this error was so small that it could safely be neglected.

As the intention was not only to ascertain the density of oxygen and nitrogen at their respective boiling-points under atmospheric pressure, but also under diminished pressure, experiments were made with nitrogen at ordinary temperatures and at pressures varying from about one-sixth of an atmosphere to ordinary pressures, in order to find the range of variation in the results with the 316 c.c. flask to be used in the subsequent low temperature experiments. In Table I. are details of these observations, and the results reduced according to the ordinary gaseous laws.

In reducing these observations the following corrections were involved:—A correction of 0·0015 grm., due to imperfect exhaustion of the flask A while being weighed as empty; the correction due to the neck of the flask between the mark and the stopcock was negligible; the volume of the flask when corrected for temperature was 315·973 c.c., and when it was necessary to correct further for excess of external over internal pressure, the values were as given in Column V; in the last four experiments this correction varied from about 0·059 to 0·073 c.c.

The first three experiments give a mean value of 1·260 grms., at standard temperature and pressure, as the weight of a litre of nitrogen. This is about a quarter per cent higher than the accepted value of 1·257. The extreme variation in the individual experiments is about half a per cent. The average value of the results under about one-third of an atmosphere is 1·266 grms.; the tendency under the low pressures being to make the density half a per cent higher. Considering that in the actual low-temperature experiments the mass of gas to be weighed would be at least three times greater, it was inferred that in spite of difficulties of manipulation and corrections the results might be anticipated to lie within a half per cent of the true value.

Table II. gives the results of six experiments made on the density of the vapour of oxygen at its boiling-point taken as 90·5° absolute, and under atmospheric pressure, where p, p_1, T_1, a, b, are the same symbols as used in the previous table, d is the calculated density at 90·5° absolute and 760 m.m., and v is the specific volume $1/d$.

In these reductions the following corrections were in-

* A Paper read before the Royal Society, January 30, 1902.

TABLE I.—*Density of Nitrogen, Ordinary Temperature.*

No.	p_1.	T_1.	p.	a.	b.	V.	d.	d_0.
	M.m.	°	M.m.	Grm.	Grm.			
1.	742·0	15·5	742·0	0·37	−0·0022	315·973	0·001168	0·001265
2.	742·0	15·5	742·0	0·3668	−0·001	315·973	0·001162	0·001258
3.	742·0	16·0	742·0	0·367	−0·0015	315·973	0·001161	0·001259
4.	751·0	16·5	281·3	0·371	−0·2328	315·913	0·000442	0·001265
5.	751·0	16·5	284·2	0·371	−0·231	315·913	0·000447	0·001269
6.	751·0	17·0	268·5	0·371	−0·239	315·911	0·000422	0·001269
7.	753·0	17·5	176·2	0·3705	−0·2850	315·899	0·000275	0·001262

Where p_1 = barometric pressure at the time of observation,
T_1 = temperature of the room at the time of observation,
p = pressure of the nitrogen vapour in the flask during the experiment,
a, b = as defined before,
V = the volume of the flask corrected for temperature and compression,
d = the calculated density at T_1° and pressure p,
d_0 = the value of d reduced to 0° and 760 m.m. by the ordinary gaseous laws.

TABLE II.—*Density of Oxygen Vapour at its Boiling-point.*

No.	$p=p_1$.	T_1.	a.	b.	d.	v.
	M.m.	°	Grm.	Grm.		
1.	775·5	15·5	0·388	1·0225	0·004402	227·15
2.	770·3	16·5	0·3865	1·015	0·004403	227·07
3.	771·0	17·5	0·3875	1·0245	0·004432	225·60
4.	771·8	17·5	0·3875	1·0260	0·004432	225·59
5.	775·0	16·0	0·388	1·028	0·004422	226·12
6.	774·2	16·5	0·3875	1·028	0·004425	225·97

TABLE III.—*Density of Oxygen Vapour at its Boiling-point under Diminished Pressure.*

No.	p_1.	T_1.	p.	a.	b.	V.	d	v.
	M.m.	°	M.m.	Grm.	Grm.			
1.	741·8	18·5	287·0	0·3695	0·1315	314·340	0·001588	625·74
2.	741·8	18·5	281·5	0·3695	0·1345	314·339	0·001607	622·01
3.	746·5	20·0	279·0	0·3690	0·1290	314·338	0·001588	629·41
4.	746·5	20·0	310·2	0·3690	0·1880	314·342	0·001776	562·95
5.	746·5	19·0	159·4	0·3690	0·0850	314·323	0·000907	1101·46

TABLE IV.—*Density of Nitrogen Vapour at the Boiling-point of Oxygen.*

No.	$p=p_1$.	T_1.	a.	b.	d.	v.	T.
	M.m.	°	Grm.	Grm.			°
1.	771·8	17·5	0·3875	0·8555	0·0039021	256·27	(90·5)
2.	773·0	17·0	0·385	0·8555	0·003885	257·39	(90·5)
3.	777·2	16·0	0·3885	0·9575	0·004192	238·53	84·05
4.	777·3	16·5	0·3885	0·9500	0·004168	239·90	84·54
5.	777·3	16·5	0·3885	0·942	0·004143	241·34	85·04
6.	777·3	16·5	0·3845	0·9235	0·004073	245·49	86·50

volved:—0·002 grm. due to imperfect exhaustion of A flask while being weighed as empty; 0·0002 grm. due to the contents of the neck of the flask between the mark and the stopcock not being at 90·5°; the volume of the flask up to the mark contracted at 90·5° to 314·398 c.c.; no correction was necessary for compression.

Thus the mean weight of 1 litre of oxygen vapour at 760 m.m. and 90·5° absolute is 4·420 grms., and the specific volume is 226·25 c.c. If the first two experiments are eliminated on the assumption that the proper equilibrium of temperature had not been attained, the average weight per litre would become 4·428 grms., and the specific volume 225·82.

Taking Regnault's density of oxygen at 0° and 760 m.m., the density at 90·5° in the ordinary way would be 0·0043137, and the specific volume 231·82 c.c. Thus the volume given by the ordinary gaseous laws is 1·0246 times the average observed volume; or we may put it that pv is diminished at the boiling-point of oxygen by 2·46 per cent. Again, while the ratio of the absolute temperatures is 3·017, the ratio of the densities is 3·091.

Behn (*Ann. der Physik.*, vol. i., 1900, "Sublimationswärme der Kohlensäure und die Verdamfungswärme der Luft") has determined by an indirect method the specific volume of oxygen, and finds the value 358, which is nearly 60 per cent greater than the volume found by the direct method. The mode of procedure he adopts is to ascertain directly three quantities out of four in the ordinary thermodynamic equation, correlating latent heat, temperature, increment of pressure to temperature, and specific volume, thereby deducing the unknown quantity. Now, of the three experimental values required, one, viz., increment of pressure to temperature, can only be got by calculation from the vapour-pressure curve, and much depends upon the accuracy of this value. Accepting Estreicher's vapour-pressures for liquid oxygen below its boiling-point as the most reliable, a Gibbs equation gives the increment per degree near 740 m.m. pressure as 78·67 m.m. mercury pressure, or 106·93 grms. per square c.m. This value, taken along with the latent heat found by Behn, and the boiling-point, gives, when inserted in the thermodynamic equation, a specific volume of 223·55, which is within less than 2 per cent of the value found by the direct-density determinations.*

Further experiments were made on oxygen vapour at 90·5° and under reduced pressures. These experiments

* In the same way Behn's specific volume of carbonic acid would be contradictory of my proof that a constant-volume gas-thermometer filled with carbonic acid at about atmospheric pressure gives a very accurate value of its own boiling-point. Assuming the ordinary gaseous laws, the specific volume ought to be 361·6 instead of 423 given by Behn. Now my value of the increment of pressure to temperature at the boiling-point is 62·84 m.m. mercury pressure, and this, along with the values used by Behn in the thermodynamic equation, gives 363 as the specific volume. This comes much nearer the anticipated value of the constant.

and their results are given in Table III. The same symbols are used as in the preceding tables, except that d is the calculated density at 90·5° absolute and pressure p, and v is $1/d$. The corrected volume of the flask is entered under V.

In reducing these observations the following corrections were involved:—A correction of 0·0014 grm. to 0·0015 grm. due to imperfect exhaustion of the A flask while being weighed as empty; the correction due to the neck of the flask between the mark and the stopcock not being at 90·5° amounted to 0·00003, 0·00007 grm., and was practically negligible; the volume of the flask, which, when corrected for temperature, was 314·398 c.c., had to be further corrected for excess of external pressure over internal pressure by amounts varying from 0·0551 c.c. to 0·0742 c.c. If the first three experiments are averaged (the pressures being so near), the weight of a litre of oxygen at 90·5° absolute under a pressure of 282·5 m.m. would be 1·5982 grms. The ratio of this density to the value previously found for one atmosphere pressure, viz., 4·42 grms., is 2·765, and the ratio of the pressures is 2·690. It appears that the ratio of the change of density of the vapour of oxygen at 90·5° absolute, under variable pressure, is greater than the ratio of the change of pressure. It is clear, however, that it would be necessary to work upon a larger scale in order to get satisfactory vapour densities at low temperatures under pressures below that of the atmosphere.

Table IV. gives the observations on the density of nitrogen vapour at the boiling-points of liquid oxygen and liquid air respectively; the first two were made in oxygen, the last four in air. The symbols used are the same as before, except that d is the calculated density at T° absolute and 760 m.m., and v is $1/d$.

In reducing these observations, the following corrections were involved; a correction of 0·002 grm. due to imperfect exhaustion of the A flask, while being weighed as empty; the correction due to the neck of the flask between the mark and the stopcock not being at temperature T amounted to 0·00014 grm., and was practically negligible; the volume of the flask, which only required to be corrected for temperature, was 314·398 c.c.

Experiments 1, 2 were made with liquid oxygen taken to be at temperature 90·5° absolute. The experiments 3, 4, 5, 6 were made in one and the same sample of liquid air, with rising temperature. For these temperatures, obviously only a few degrees below the boiling-point of oxygen, the ordinary gaseous laws may be held to apply, in order to determine their values. Thus we may employ the formula—

$$T = v \frac{90{\cdot}5}{256{\cdot}833},$$

where 256·833 is the mean of the volumes of Nos. 1, 2, to get the temperature of the last four experiments. The values thence obtained are entered in column T.

The first two experiments made with liquid oxygen give a ratio of the nitrogen densities from my own values of 3·088, the absolute temperature ratio being 3·017; my values for the ratio of the oxygen densities for the same range of temperature being 3·091 as previously deduced. We may safely assume that if the density of nitrogen were observed at its boiling-point it would deviate as much from the ordinary gaseous laws as oxygen. Further, the specific volume of nitrogen at its boiling-point of 78° absolute would from the above formula be 221·3 as compared with 226·2, the similar value found for oxygen.

The general inference to be drawn from these preliminary experiments is that reliable vapour densities may be determined at very low temperatures. There seems to be no reason why the vapour density of hydrogen at its boiling-point should not be accurately ascertained; only, as in this case the internal pressure in the weighing flask would amount to nearly 15 atmospheres, it would be advisable to construct the flask of some metal or alloy. A flask of the size used in the oxygen experiments filled with the vapour of hydrogen at its boiling-point would be equivalent in weight to between 4 and 5 litres of hydrogen at the ordinary temperature and pressure, and such an amount of material ought to give density results at the boiling-point of hydrogen of considerable exactness, notwithstanding the great manipulative difficulties that would necessarily be involved in the execution of such a determination at 21° absolute.

IMPROVED ELECTRIC FURNACE FOR LABORATORY USE.

By SAMUEL AUCHMUTY TUCKER
and
HERBERT R. MOODY.

ELECTRIC furnaces hitherto described have been found by the writers to possess certain disadvantages for experimental purposes. This led them to devise the form of furnace herein described which it is thought is a convenient piece of apparatus to build, and will be found to be well adapted to a variety of work in the laboratory.

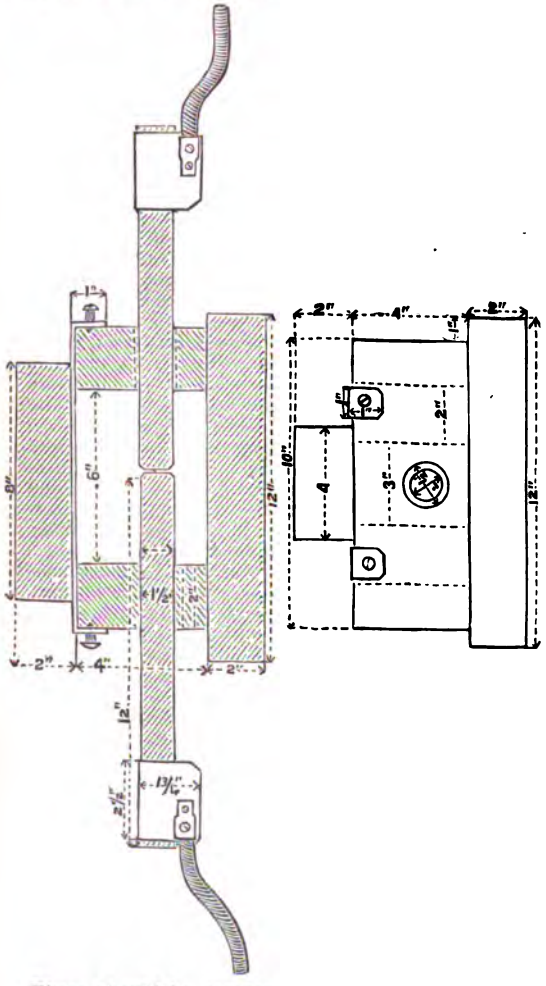
The Moissan type of furnace is now well known to every one, but it is troublesome in several ways. If built from chalk blocks in the rough, it takes both skill and time to cut them to the exact form required; it is then necessary to dry the blocks very thoroughly, otherwise they will crack in all directions, and render the furnace useless immediately. A perfectly dry furnace supported by metallic bands will generally crack to some extent, and it is seldom possible to put the furnace in use a second time. The form of furnace described by one of us (*American Electrician*, xi., 408), which simply consists of a graphite crucible forming one pole, the other being a carbon rod supported vertically with an arrangement for raising and lowering it at will, is useful for some purposes, but there is too much exposure to the air for many operations, and the material of which the crucible is composed is likely to introduce undesirable impurities during the fusion. The operator is exposed to very intense radiant heat, which interferes considerably with its use for any lengthened period. The present form of furnace is after the Moissan type, and is composed of carbon bricks 12 inches by 4 inches by 2 inches, luted together with Dixon stove paste. The sides of the furnace were of 6-inch brick, thus making a working space of about 6 by 4 by 2 inches. This space could, of course, be increased or diminished at will according to the charge used. The whole was then clamped, and held firmly together by iron cross bars provided with adjusting screws at each end, these bars being insulated from the body of the furnace by strips of asbestos. The end bricks were perforated with 1·5-inch holes containing a collar of asbestos or a small cylinder of clay, through which the electrodes of carbon passed (1 inch by 12 inches) into the furnace chamber. Connection was made with these electrodes by copper sleeves lined with copper gauze, and tightened with set screws, which at the same time carried copper lugs which held the flexible cables.

The tendency to overheat those portions of these electrodes outside of the furnace, and consequent wasting away, and also the tendency to melt the copper connections, was overcome by either one of two expedients; that is, by the use of the water-jacket, or by heavily copper-plating the carbons. This latter did not furnish quite as perfect a protection, but it was somewhat less troublesome than the former, and was, consequently, the one most used.

The construction of this furnace gives it considerable durability; so much so is this the case, that only three sets of brick were used throughout the year, in which almost a hundred runs were made. The greatest deterioration comes from the oxidation of the outer surface of the carbon bricks, and this could be largely avoided by a suit-

able covering of fire-bricks. Frequently ten or twelve runs could be made without dismantling the principal parts, and the substitution of new asbestos collars serves to make everything ready for the next run.

In this type of furnace the desired current may be readily maintained throughout the entire fusion, and this is not easily done with a crucible furnace where variations are unknowingly introduced by the constantly changing distance between the poles. This form of furnace may be used with double arc on 110 volt circuit with considerable economy. For example, with a single arc furnace and with a polar separation of 18 m.m. and enough resistance in circuit, the following reading was obtained:—125 ampères, 70 volts, and, consequently, 8750 watts.



These should be compared with the following results obtained while using the double arc furnace. Here all the resistance was cut out, and then the instruments showed 175 ampères and 90 volts. A few moments after this the reading was 125 ampères, and 100 volts or 12,500 watts.

The only difficulty in using this type is to prevent the extinction of the arcs, but that trouble is far from insurmountable. The dimensions given for the furnace are suitable for currents up to 300 ampères, at 70 volts. For work requiring more power than this it is necessary to increase the size throughout and use electrodes of 2-inch diameter.

If the luting has been carefully done the furnace will be sufficiently gas-tight to permit a fusion to be carried on in an atmosphere of any desired gas, the gas being introduced through an annular electrode. This furnace is adapted to almost all kinds of fusion processes, and has been used in the greatest variety of work.—*Journal of the American Chemical Society*, xxiii., No. 7.

NOTE ON SOME MODIFIED FORMS OF PHYSICO-CHEMICAL MEASURING APPARATUS.*

By ALLERTON S. CUSHMAN.

A. *A Convenient Arrangement of the Kohlrausch-Ostwald Conductivity Cell.*

In working with the usual form of Kohlrausch-Ostwald conductivity cell the writer has experienced some difficulty in arranging the electrodes so that they may be easily adjusted at any required distance one from the other, while, on the other hand, when once set to a position the constant for the cell may be depended upon. The growth of physico-chemical methods of investigation has been so sudden that the manufacturers of instruments of precision

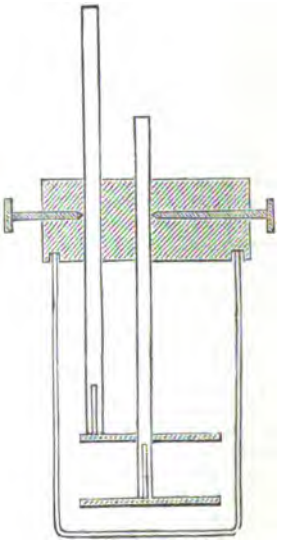


Fig. 1.

have not kept pace with the requirements of modern research. The physical chemist, therefore, has been obliged to depend largely on "home-made," and too often very imperfect, apparatus. It is not uncommon in many laboratories to see this important and delicate measuring instrument arranged in such a way that the electrodes are most insecurely fastened into place by means of splashes of sealing-wax, or, as is the case with the cells ordinarily supplied by the dealers, the mere friction of the glass supports of the electrodes against the holes in the ebonite cover through which they pass, is depended upon to insure the constancy of the cell.

In Fig. 1 of the accompanying illustrations is shown an arrangement of the cell which very admirably meets every requirement. The glass tubes into which the electrodes are fused fit snugly, but without binding, the holes in the heavy ebonite cover, which is in cross-section in the cut. The brass set-screws as shown are furnished with ebonite

* Contribution from the Chemical Laboratory of Bryn Mawr College. From the *Journal of the American Chemical Society*, xxiii., No. 7.

tips, which bear gently on the glass supports and remove the danger of contamination of the electrolyte by any accidental wetting of the screw points. A cell of this description which has been in frequent use for more than a year has not changed its constant. The electrodes are made of platinum, of nearly a millimetre in thickness, and are secured to the glass supports with such care that they are quite immovable.

B. *On a Modified Form of the Ostwald Burette-Calibrator.*

The usual Ostwald burette-calibrator has the general form shown in Fig. 2, without the etched scale upon the pipette stem. It is calibrated to deliver exactly 2 c.c. between two etched marks, and the deviations of the burette scale from the truth are then found by a series of deliveries from the pipette. The initial standardisation of the pipette requires a number of careful and difficult

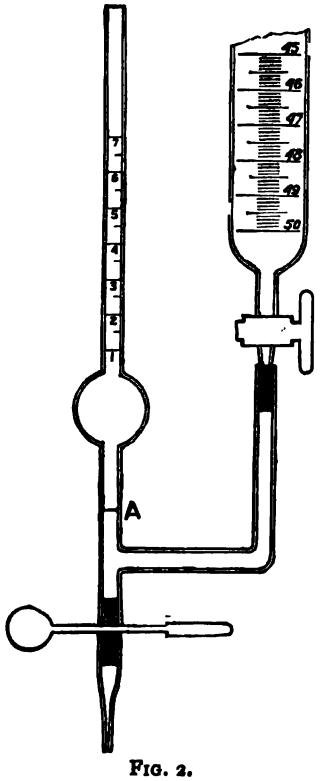


FIG. 2.

weighings of water. In order to avoid this standardisation the writer has used a pipette with a scale etched upon the stem as shown in the cut. It is only necessary to take care that when the pipette is filled to the mark etched at A that about 2 c.c. more will put the meniscus somewhere near the middle of the pipette scale. Starting with the burette filled to the zero reading and the lower meniscus coinciding with the mark at A, the first 2 c.c. of the burette scale are delivered into the pipette, and the pipette-scale reading noted. The pipette is then emptied to A, and the next 2 c.c. of the burette delivered. This operation is repeated down the length of the burette. One set of readings can be made starting from the zero point of the burette and another set starting from the 1 c.c. graduation mark. In order to apply the corrections it is only necessary to standardise the pipette scale against the burette scale by a few fractional fillings of the pipette. In applying the corrections any of the ordinary methods of calibration may be employed, such as assuming the first 2 c.c. of the burette correct, and then distributing the deviations from the truth. For most laboratory work exceedingly small deviations are negligible, but the fact that a serious deviation from the truth may introduce a constant error into a series of observations, makes a rapid and easy method of becoming acquainted with the variations of burette scales of great value. This instrument permits this to be done with but little expenditure of time, and without the necessity of making a single weighing. Bad burettes can be immediately condemned, and those of sufficient accuracy for the work in hand selected. In case absolute corrections are demanded, the pipette can be standardised by the method of weighing much more easily than one that is not provided with an etched scale on the pipette stem. Of course the pipette must be made scrupulously clean with a chromic-sulphuric acid mixture before it is used.

A BIBLIOGRAPHY OF STEEL WORKS ANALYSIS.

PART XI.

By HARRY BREARLEY.

(Continued from vol. lxxxiv., p. 250).

COPPER.

THIS section extends to the end of 1900. The items are arranged under the following heads:—

I. SEPARATIONS FROM OTHER METALS.
II. GRAVIMETRIC ESTIMATIONS.
III. VOLUMETRIC ESTIMATIONS.
  a. Cyanide Process.
  b. Iodide Process.
  c. Other Processes.
IV. COLORIMETRIC ESTIMATION.
V. DETECTION.
VI. MISCELLANEOUS.

I. SEPARATIONS FROM OTHER METALS.

Iron (see also 1495, 1537, 1541, 1608).—

1472. LOEWE (*C. N.*, i., 119).—After precipitating with AmHO, the $Fe_2(HO)_6$ cannot be *washed* free from Cu; it must be dissolved and re-precipitated.

1473. GALETTI (*C. N.*, xx., 248).—Precipitate iron with AmHO, add excess acetic acid, and again excess AmHO followed by acetic. This procedure prevents adhesion of Cu to precipitated iron. Cu titrated with ferrocyanide.

1774. MERRICK (*C. N.*, xxvi., 115).—Decompose pyrites with HNO_3, evaporate, and dissolve Cu from residue with dilute AmHO.

1475. BREARLEY (*C. N.*, lxxviii., 14).—Iron can be precipitated from neutralised solutions as basic chromate perfectly free from copper, and Cu estimated as in 1569.

1476. FLAJOLOT (*C. N.*, ix., 193).—Precipitate Cu with hypo. The precipitate is free from Fe, Zn, Co, Ni, and P, but not As or Sb. Estimate Cu with KCN.

1477. CARNOT (*C. N.*, liii., 172).—As 1476. Precipitated Cu_2S ignited with S in H and weighed.

1478. VORTMANN (*Z. C. S.*, lxvi., ii., 35).—AmHO solutions of Cu electrolysed without filtering off precipitated Fe.

1479. FERNBERGER and SMITH (*Z. S. C. I.*, 1900, 53).—Electro-separations of Fe, Cr, Zn, Ni, Co, Mn, &c., from Cu, using solutions of the phosphates in phosphoric acid.

1480. *Aluminium.*—HUNT, CLAPP, and HANDY (*C. N.*, lxv., 225).—Cu precipitated with H_2S from acid solution of metallic Al, evaporated with HNO_3, and weighed as CuO.

Zinc (see also 1479, 1518, 1524, 1541, 1543).—

1481. Wittstein (*C. N.*, xix., 156).—Zn dissolved from precipitated sulphides with dilute H_2SO_4.

1482. Berglund (*C. N.*, xlvii., 244).—Precipitate Cu with H_2S from definitely acidified solutions.

1483. Knorre (*C. N.*, lix., 222).—Precipitate Cu with nitroso-β-naphthol, and ignite to CuO (see 25 and 26).

1484. Kaikow (*J. C. S.*, lxviii., ii., 246).—Cu precipitated with hydrazine hydrochloride and KI (see 543 and 1543).

1485. Soderbaum (*J. C. S.*, lxxii., ii., 348).—Cu is precipitated by a stream of acetylene quite free from zinc.

1486. Mawrow and Muthmann (*J. S. C. I.*, 1896, 381).—Cu is precipitated free from Zn and Cd, if no HCl is present, by adding hypophosphorous acid.

Cadmium (see 26, 1486, 1495, 1524, 1532, 1542).—

1487. Behal (*C. N.*, li., 280) and Warren (*C. N.*, lxix., 125).—Add an alkaline solution of soda tartrate and boil. Cd is precipitated.

1488. Gucci (*C. N.*, li., 55).—Precipitate Cu with ammonium benzoate from acid solution.

1489. Vortmann (*C. N.*, xlv., 57).—Hypo. precipitates Cu free from Cd.

1490. Hoffmann (*C. N.*, ii., 159).—Cd is easily dissolved from mixed sulphides with H_2SO_4.

1491. Browning (*J. C. S.*, lxvi., ii., 68).—Cu precipitated as iodide, dried, and weighed; Cd in solution.

1492. Backelandt (*J. S. C. I.*, 1886, 510).—Cd is precipitated with KHO in the presence of glycerol. Cu is precipitated from the filtrate with glucose and weighed as CuO.

1493. Wells (*C. N.*, lxiv., 294).—To neutral solution add hypo. until all colour goes; then precipitate Cd with Na_2CO_3 and filter off. On acidifying filtrate, Cu is precipitated.

1494. Bornemann (*C. N.*, lxxxi., 53).—Cu precipitated from hot HNO_3 solutions with oxalic acid.

Nickel (see also 1479, 1537, 1541).—

1495. Rudorff (Ag, Hg, Cd) (*J. C. S.*, lxiv., ii., 305) and Smith and Moyer (Cd, Zn, Co, Fe) (*J. C. S.*, lxiv., ii., 496).—Electro-separations.

Mercury (see also 1584).—

1496. Rosenbladt (*J. C. S.*, lii., 302).—Precipitated sulphides digested with K thiocarbonate. Hg and Pd dissolved.

1497. Von Uslar (*C. N.*, lxxiii., 28).—Precipitate Hg as Hg_2Cl_2 with phosphorus acid from acidified solutions.

1498. Jannasch (*C. N.*, lxxiv., 145).—Hg volatilised from precipitated sulphides; or (*J. C. S.*, lxxvi., ii., 59), precipitated from AmHO solutions with hydroxylamine.

1499. Smith and Macauley (*J. C. S.*, lvi., 797; and lxii., 239) and Revay (Hg and As) (*C. N.*, lxxix., 194).—Electro-separations.

Lead (see also 1525, 1584).—

1500. Nissenson (*J. C. S.*, lxvi., ii., 220) and Classen (*J. C. S.*, liv., 529).—Electro-separations.

Arsenic (see also 1532, 1542, 1543, 1555, 1594).—

1501. Parnell (*C. N.*, xxi., 133).—Examines processes depending on solubility of As in Na_2S, passing chlorine through alkaline liquor containing precipitated sulphides, and heating in Cl gas and H.

1501*a*. Classen (*J. C. S.*, liv., 528).—By volatilising As from HCl solution.

1502. Jannasch (Sb and Sn) (*J. C. S.*, lxviii., ii., 89 and 462).—By volatilising in a stream of HCl gas.

1503. Gucci (*J. C. S.*, liv., 630).—Cu precipitated with KHO.

1504. McKay (*J. S. C. I.*, 1890, 822), Smith and Frankel (*J. S. C. I.*, 1890, 1067), and Schmucker (*J. S. C. I.*, 1894, 280).—Electrolytic.

Antimony (see also 1502, 1549, and 1555).—

1505. Lucas (*C. N.*, lxxix., 78).—Treat mixed sulphides with Na_2S, destroy polysulphides with H_2O_2, and electro-deposit Sb without removing CuS, as in 516.

1506. Finkener (*J. S. C. I.*, 1889, 733).—Add alkaline fluoride, and then KI and SO_2. The filtrate contains Sb_2O_5 and a little Cu, which are separated with H_2S and Am_2S.

1507. Smith and Wallace (*J. S. C. I.*, 1893, 1063).—Electro-separation.

Bismuth.—

1508. Lowe (*C. N.*, xlviii., 296).—Precipitate with NaHO, re-dissolve in glycerin, and precipitate Cu with glucose in the cold. Bi in solution.

1509. Jannasch (*J. C. S.*, lxvi., ii., 71).—Bi precipitated with AmHO and H_2O_2.

1510. Smith and Saltar (*J. S. C. I.*, 1893, 605).—Electro-separation from a citrate solution containing regulated amount of KCN.

Silver (see also 1495, 1584).—

1511. Revay (*J. S. C. I.*, 1898, 184) and Kuster and Steinwehr (*J. S. C. I.*, 1898, 466).—Electro-separations.

Selenium.—

1512. Violette (*C. N.*, xxi., 178).—Heat strongly in current of dry air; the Se is sublimed as a white crystalline ring.

Palladium (see also 1496).—

1513. Wöhler (*C. N.*, xv., 40).—Cu precipitated with SO_2 and KCNS.

Thallium.—

1514. Crookes (*C. N.*, vii., 133 and 218).—Precipitate Tl as sulphide from alkaline cyanide solution; or, precipitate metals as subiodides and dissolve out the Cu with AmHO. Metallic Cu frequently contains Tl.

II. Gravimetric Estimations.

1515. Storer (*C. N.*, iii., 70).—Deposit Cu from H_2SO_4 solutions with iron, and heat in nitrogen before weighing to destroy organic matter.

1516. Kern (*C. N.*, xxxi., 76).—Cu-Sn-Zn alloys, after decomposition, are precipitated as metals on Fe, Zn, &c.

1517. Villiers (*C. N.*, lxviii., 263).—Precipitate Cu with Mg, wash with alcohol, dry at 100° C., and weigh. An alloy is precipitated if Zn is present.

1518. Warren (*C. N.*, lxi., 136; and lxxi., 92).—Decompose Zn-Cu alloy with strong H_2SO_4, precipitate Cu with Mg, and weigh. After adding soda acetate, Zn may be precipitated similarly.

1519. Shengle and Smith (*C. N.*, lxxxi., 134).—Cu precipitated by zinc always contains more or less of the latter metal.

1520. Mohr (*C. N.*, vi., 229).—Decompose ore with H_2SO_4, evaporate, and boil dry residue with water. Cu and a little Fe dissolve; precipitate Cu with Zn and wash out of contact with air.

1521. Sutton (*C. N.*, vi., 275).—A score of common elements have no influence on 1520.*

1522. Lowe (*C. N.*, ii., 191).—Cu is completely precipitated by a slight excess of oxalic acid.

1523. Gibbs (*C. N.*, xvii., 172).—Cu is completely precipitated by boiling with small excess of Na_2CO_3. The metal reduced in H is free from alkalis (see also Field, *C. N.*, ii., 279).

1524. Warren (*C. N.*, lxiii., 193).—Precipitate with glucose from solution containing NaHO and Rochelle salt and weigh as CuO. Zn and Cd perfectly separated. For various means of esti-

* Simple replacement of copper by either Zn, Mg, or Fe is found in no case to be exactly true. Clowes's paper (*C. N.*, lxxviii., 155) may be taken as a type of many others.

mating the suboxide precipitated by glucose, see *C. N.*, xliv., 262.

1525. Jannasch and Lezcinsky (*C. N.*, lxix., 7).—Separate Pb from AmHO solution with H_2O_2. Precipitate Cu in H_2S and heat in air which has passed through Am_2CO_3, so as to decompose any $CuSO_4$ formed.

1526. Millon and Commaile (*C. N.*, viii., 100).—Copper always escapes with the nitrous vapours on heating $Cu(NO_3)_2$. Weigh as Cu after reducing in H.

1527. Wegscheider (*J. C. S.*, lxvi., ii., 31).—Particulars of temperature and nature of atmosphere to be attained when Cu is estimated as cuprous sulphide (see also 1578).

1328 Debray and Joannis (*C. N.*, l., 234 and 248).—A temperature high enough to melt or even frit CuO partly decomposes it.

1529. Ulrici (*C. N.*, xx, 118).—Ignited precipitate formed with H_2S is weighed as though Cu_2S, but really it is always a mixture of CuO and Cu_2S.

1530. Holthof (*C. N.*, lxi., 193).—Precipitated sulphide ignited while moist passes to oxide completely. Dried precipitates contain SO_3 after ignition.

1531. Stickney (*J. C. S.*, lxx., ii., 523).—Cu_2S is reduced to copper when heated in contact with a Bunsen flame.

1532. Nissenson and Neumann (*J. C. S.*, lxx., ii., 450).—Cu precipitated with hypo. is oxidised completely to CuO in the muffle. Any As is expelled. Cd does not interfere. Also Willenz (*J. C. S.*, lxxviii., ii., 315).

1533. Troilius (*J. I. S. I.*, 1883, 367 and 710).—Precipitate H_2SO_4 solution of steel with hypo.; re-dissolve and re-precipitate, and ignite moist precipitate to CuO.

1534. Reinhardt (*J. I. S. I.*, 1889, ii. 481).—Reduce ferric solution with hypophosphite and precipitate Cu with H_2S. H_2SO_4 (3 : 1) dissolves basic pig without attacking contained copper.

1535. Ziegler (*J. S. C. I.*, 1893, 377).—Ammonium sulphocarbonate, which is more agreeable in use than H_2S, precipitates Cu from FeO solutions of steel.

1536. Reis (*J. I. S. I.*, 1891, i., 441).—HCl solution of steel is treated with H_2O_2 to bring any undissolved Cu into solution; then reduced as 1534, and Cu precipitated with sulphocarbonate and ignited to oxide.

1537. Klein (*J. S. C. I.*, 1887, 836).—Add ammonium dithiocarbamate to hot HCl solution. Ignite precipitate with S in H and weigh as Cu_2S. Fe, Mn, and Ni do not interfere.

1538. Parry and Morgan (*C. N.*, lxvii., 259).—Precipitate from FeO solution with H_2S or hypo., and re-precipitate with NaHO or ignite at once to CuO. Also, digest nearly dried residue of 20 to 50 grms. of steel with AmHO and compare blue colour with standards.

1539. Nahusen (*C. N.*, lx., 133).—Decompose pyrites with $H_2SO_4+HNO_3$, precipitate with H_2S, dissolve out As and Sb with Na_2S, and estimate Cu as sulphide.

1540. Bloxam (*C. N.*, xi., 196).—On digesting the sulphide with Am_2S, a portion of the Cu dissolves as $Cu_2(NH_4)_2S_7$.

1541. Gibbs (*C. N.*, xvii., 160).—Precipitate from H_2SO_4 solution with Mg hypophosphite, ignite in H, and weigh as Cu. Fe, Mn, Ni, and Zn do not interfere; Sb, As, HCl, and HNO_3 do.

1542. Soderbaum (*J. S. C. I.*, 1897, 563).—Cu precipitated from alkaline solution with acetylene. Dissolve acetylide in HNO_3, filter from C, evaporate, and ignite to CuO. *J. C. S.*, lxxiv., ii., 191.—As and Cd are separated from Cu in this way.

1543. Jannasch and Biedermann (*C. N.*, lxxxii., 282).—Hydrazine sulphate precipitates metallic Cu from NaHO solutions free from Zn, As, and nearly Sn. Cu weighed as CuO.

1544. Tamm (*C. N.*, xxiv., 91).—Precipitate as sulphocyanide (see 1584), digest with Am_2S, and weigh as Cu_2S. Cu may be separated as CuCNS from all other elements.

1545. Millon and Commaile (*C. N.*, vii., 218).—Process for estimating Cu (or Ag) or mixtures of CuO and Cu_2O is based on the fact that AmHO solutions of Cu_2O precipitate Ag from silver solutions.

1546. Gibbs (*C. N.*, xi., 173).—Precipitation with Zn accurate (see 1519). Suggests electro-deposition from sulphate solutions.

1547. Luckow (*C. N.*, xix., 221).—Dissolve roasted ore in acid and electro-deposit Cu. Behaviour of associated elements.

1548. Hampe (*C. N.*, lxvii., 103).—Small quantities of Sb are always electro-deposited with Cu. So also is Bi (*C. N.*, lxviii., 51). The Bi may be removed as oxychloride (Stahl, *J. C. S.*, lii., 529). See also Smith and Saltar (*J. C. S.*, lxiv., ii., 495), who say Pb is also deposited.

1549. Smith and Wallace (*J. C. S.*, lxiv., ii., 495).—No Sb is deposited with the Cu from AmHO tartaric solutions.

1550. Chassy (*C. N.*, lxx., 97).—On electrolysis a heated $CuSO_4$ solution deposits Cu_2O.

1551. Merrick (*C. N.*, xxxiii., 111).—Can determine 0·0001 grm. Cu by depositing on Pt.

1552. Mackintosh (*C. N.*, xliv., 279; and xlv., 101).—Deposition from nitro-citric acid solutions too high; from HNO_3 alone difficult to precipitate all the Cu. $CuSO_4$ solutions best.

1553. Rudorff (*J. C. S.*, lxiv., ii., 93).—From faintly HNO_3—or, if Cl is present, from AmHO—solutions. Soda acetate is added before stopping the current.

1554. Drossbach (*J. C. S.*, lxiv., ii., 93).—Electrolyse AmHO solutions only until the Cu is precipitated. Hg, Cd, Pb, and Ag are the only interfering elements.

1555. Œttel (*J. C. S.*, lxviii., ii., 139).—Best deposited from AmHO solution if Cl, As, or Sb are present. Pb, Bi, Hg, Cd, and Ni interfere.

1556. Classen (*J. C. S.*, xlviii., 1095; and lvi., 77) and Wagner (*J. C. S.*, lxxii., ii., 520).—Deposit from oxalate solutions at 70—80° C.

1577. Hollard (*C. N.*, lxxvi., 31).—Electrolytic methods for complete analysis of brass and bronze.

1558. Brand (*J. S. C. I.*, 1889, 1011).—From pyrophosphate solutions.

1559. Wolman (*J. C. S.*, lxxiv., ii., 50).—Best results obtained by deposition from H_2SO_4 or HNO_3 solutions.

1560. Moore (*C. N.*, liii., 209).—Deposits from alkaline KCN solutions at 70°.

(To be continued)

Examinations in Modern Languages.—The Society of Arts is endeavouring to extend its modern languages examinations, so as to make them include a *vivâ voce* test. The written examinations are now carried on at 300 centres throughout the United Kingdom. A notice has been sent out to all these centres, to the effect that a *vivâ voce* examination will be held at any centre where 24 candidates can be collected, at a fee of 2s. 6d. a head. The Society's examinations include all modern languages of commercial importance, but at present *vivâ voce* examinations will only be held in French, German, and Spanish. The tests are to include dictation, reading, and conversation.

EXPERIMENTS RELATIVE TO THE CONSTITUTION OF PECTOLITE, PYROPHYLLITE, CALAMINE, AND ANALCITE.*

By F W. CLARKE and GEORGE STEIGER.

(Concluded from p. 66).

Analcite (continued).

In order to discuss the constitution of analcite, let us recur to the analysis of the mineral itself. It is at once evident from the comparison made on a preceding page that our sample of the mineral varies notably in composition from the requirements of theory. The silica is 2·5 per cent too high, while alumina and soda are correspondingly low. No probable impurity and no presumable errors of manipulation can account for so great a divergence. If we consult other analyses, as we find them tabulated in manuals like those of Dana and of Hintze, we shall find other cases resembling this, and also examples of variation in the opposite direction, with silica low, and an apparent excess of bases. Most analcite gives quite sharply the metasilicate ratios required by the accepted formula; but the variations from it are large enough, common enough, and regular enough to command attention. The analyses are not all covered by the recognised theory, and the apparent irregularities are not fortuitious, but are systematic in character.

One explanation of the seeming anomalies is simple and clear. If analcite, instead of being a metasilicate, is really a mixture of ortho- and tri-silicate, then all of the analyses become intelligible. In most cases the two salts are commingled in the normal ratio of one to one; but in our analcite the trisilicate predominates, while in some other samples the ortho-salt is in excess. All reduce alike to the simple expression—

$$NaAlX.H_2O,$$

in which X represents $nSiO_4+mSi_3O_8$; a formula which agrees with evidence from various other sources.

For example, analcite may be derived in nature either from albite, $AlNaSi_3O_8$, or nephelite, $AlNaSiO_4$, and, on the other hand, alterations of it into felspars have been observed. Its closest analogue, leucite, has yielded pseudomorphs of orthoclase and elæolite; while leucite and analcite are mutually convertible each into the other. The evidence of this character, the evidence of relationship between analcite and other species, is varied and abundant, and the simplest conclusion to be drawn from it is that which has been given. Every alteration, every derivative, every variation in the composition of analcite points to the same belief. The consistency of the data can not well be denied.

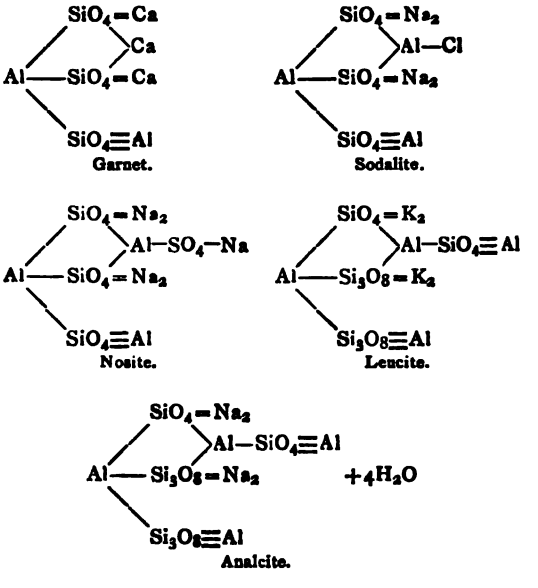
In the case of a normal analcite—that is, one which conforms to the usual empirical formula—the expression which best represents these relations is—

$$Al_4Na_4(SiO_4)_2(Si_3O_8)_2.4H_2O;$$

and this accords with the minimum molecular weight as determined by the study of our ammoniated residue. Structurally, this is comparable with the formulæ of garnet, zunyite, sodalite, nosite, and leucite; all of which are also isometric in crystallisation. The more important of the symbols are given in next column.

That is, analcite and leucite become members of the garnet-sodalite group of minerals, and their relations to nephelite, albite, prehnite, natrolite, &c., natural and artificial, are perfectly clear. In analcite there may be admixtures of strictly analogous ortho- or tri-silicate molecules; but these remain to be separately discovered.

Now, these formulæ are not ultimate verities to be blindly accepted. They are simply expressions which represent composition, and a wide range of established relationships, and which serve a distinct purpose in the correlation of our knowledge. Properly used, with due recognition of their limitations, they are helpful, and suggest possibilities of research; misused, they may become mischievous. They now satisfy all known conditions, and that is a sufficient warrant for their existence.



* *Bulletin of the United States Geological Survey*, No. 167.

ON PYRITE AND MARCASITE.*

By H. N. STOKES.

(Continued from p. 65).

IV. Data for Pyrite.

The following table gives duplicate determinations of p, made upon well-crystallised samples of pyrite free from visible impurities, but otherwise selected at random:—

Determinations of p *made upon Samples of Pyrite.*

No.	Locality.	Density.	p.	Mean value of p.
1.	Unknown	—	60·1 59·9	60·0
2.	Custer County, Colo. ..	5·018	60·4 59·7	60·0
3.	Roxbury, Conn.	5·023	61·2 60·9	61·0
4.	Leadville, Colo.	—	60·2 60·2 60·4	60·3
5.	Old Jordan mine, Utah..	5·041	60·5 61·1	60·8

Mean value of p 60·4.

No. 1. A large cube, with slight tarnish and splendent conchoidal fracture. Traces of silica, copper, and arsenic.

No. 2. Octahedron, with fracture on peripheral part conchoidal, on the interior somewhat uneven or granular. Slight trace of copper,

No. 3. Combination of cube and octahedron, with conchoidal fracture. Traces of copper and arsenic.

No. 4. Cubes. Traces of silica and copper.

No. 5. Cubes, splendent conchoidal fracture. Trace of copper.

* From the *Bulletin of the U.S. Geological Survey*, No. 186.

Oxidation Coefficient of Pyrite.

From the above figures it appears that the oxidation coefficient of pyrite is not likely to vary more than 0·6 per cent on duplicate determinations, and that it may vary about 1 per cent in typical specimens, which do not contain more than traces of impurities that can influence the result. Whether this difference is due to the chemical action of the impurity on the solvent, to its electrochemical action on the pyrite, or to slight physical differences in the pyrite itself, can not be decided at present. The oxidation coefficient of pure pyrite may be assumed to be 60·4. Figures higher than 61 or lower than 60 may be taken to indicate greater contaminations, which can usually be detected by analysis, and the influence of some of which it is pointed out below.

V. Data for Marcasite.

The following table gives the values of p (mostly in duplicate) for various samples of marcasite, which were selected as being free from visible impurity and showing characteristic crystallisation. Other specimens, which were found to contain pyrite, are described in a later section.

Values of p *for Various Samples of Marcasite.*

No.	Locality.	Density.	p.	Mean value of p.
6.	Dover Cliffs, England ..	4·881	16·3	16·3
7.	Galena, Ill.	4·891	16·9 16·1	16·5
8.	Galena, Ill.	—	16·5 26 7	16·6
9.	Linden mine, Wis. ..	4·901	16·3 16·9	16·6
10.	Galena, Ill.	4·886	17·4 17·3	17·4
11.	Hazel Green, Wis. ..	4·896	18·1 17·5	17·8
12.	Weardale, England ..	4·880	18·0 18·0	18·0
13.	Cornwall, England ..	4·878	18·7 18·3	18·5
14	Webb City, Mo.	4·887	18·6 18·8	18·7

No. 6. This is the specimen described by Julien (*Annals New York Acad. Sci.*, 1887, vol. iv., p. 172) as follows:—"No. 14, Marcasite, Dover Cliffs, England. A brilliant cluster of spearheaded crystals—broad, striated, twinned plates—embedded in light grey chalk, greyish white, and brilliant on fracture." Some of the crystals carry small pyrite crystals along the plane of twinning, which are visible only after cleaning with acid. Only those free from visible pyrite were used for the determination.

No. 7. A hollow stalactite from Galena, Ill. The inner portion is fibrous, the outer columnar, with pyramidal terminations. Tarnish, copper coloured. Colour of fresh fracture or clean surface, tin-white. It contains a trace of copper, and is free from arsenic. Only the outer columnar portion was used.

No. 8. A similar stalactite from Galena, Ill., with greenish-grey tarnish on fracture, and bronze-coloured on surface, and with decided vitriolisation.

No. 9. Curved wedge-shaped crystals of the Joplin type, from Linden mine, Wisconsin. Iridescent blue and yellow tarnish. Fracture, tin-white. The specimen carried a little galena and calcite, from which it was carefully freed by acid and by picking out. Analysis showed a trace of copper and a little lead.

No. 10. Galena, Ill. A compact mass of large acute pyramidal crystals branching out from a common axis. Tarnish, greenish-yellow. Colour on fresh fracture and on green surface, tin-white. A few crystals of chalcopyrite. Material carefully selected for determination gave traces of silica and copper, and no arsenic.

No. 11. Hazel Green, Wis. A thick crust on calcite, with distinct rhombic crystallisation. Colour, tin-white. Contains traces of lead and copper, and is free from arsenic.

No. 12. Weardale, England. Flat, deeply striated crystals, with slight bluish tarnish. Colour of clean surface, tin-white. No visible impurity was observed, and analysis showed the presence of a faint trace of copper and a little silica, and the absence of arsenic.

No. 13. Cornwall, England. Flat crystals, with slight bluish tarnish. Colour of cleaned surface, tin-white. A few of the crystals carry minute cubes of pyrite, and such were rejected. Analysis showed no arsenic, and a trace of copper and antimony. The high oxidation coefficient makes it possible that it encloses considerable pyrite, but material was wanting for a special determination of this.

No. 14. Webb City, Mo. Curved wedged-shaped crystals of the Joplin type, on sphalerite and calcite, and with a little enclosed chalcopyrite. Analysis of a sample used for the determination showed some lead and copper, which probably account for the high figure obtained.

Oxidation Coefficient of Marcasite.

From the above figures it appears that specimens of apparently pure marcasite differ somewhat more with regard to the oxidation coefficient than does pyrite. It will appear below that an admixture of small quantities of pyrite lowers the value of p, notwithstanding the fact that pyrite has a higher oxidation coefficient. It is probable that other sulphides in minute amount exert a similar effect to even a greater extent, possibly in part because of the electrochemical action which they produce. The marcasite with the lowest oxidation number is therefore not the purest. The specimen No. 12 from Weardale was the purest examined, and I incline, therefore, to adopt the figure 18 as being nearest to the oxidation coefficient of pure marcasite. Figures higher than this, as well as lower than 16·5, must be regarded as indicating contamination, either with pyrite or with another oxidisable mineral. It also appears from the examination of certain samples (Nos. 22 and 23) that marcasite may carry considerable amounts of pyrite so intimately intergrown as not to be visible upon examination of small fragments under a lens after cleaning with acid.

VI. Mixtures of Pyrite and Marcasite.

It is desirable to construct a curve which shall give the oxidation coefficients of mixtures of pyrite and marcasite. Such a curve would enable us to ascertain, in the absence of other indications, whether a given specimen contains one or the other or both of these minerals, and in the latter case to determine their relative amounts. It would enable us to test the hypothesis of Julien (*Annals New York Acad. Sci.*, 1887, vol. iv., p. 213), whose statement I quote:—

"The forms of iron pyrites occurring in nature are intimate mixtures of these three minerals; rarely of pyrrhotite, however, on account of its ready metasomatic alteration into one or the other of the triad. These common mixtures of marcasite and pyrite may originate by inclosure during crystallisation, by alteration, and by displacement, and pass progressively into complete paramorphs, well crystallised after the form of one or the other mineral.

"The latent constitution of these composite minerals is

indicated by variation in density, exactly proportionate in most cases to the amount of each constituent (compare table on p. 33), and by a similar variation in other physical properties; *e.g.*, hardness, fracture, resistance to decomposition, and even in colour, in the case of the paramorphs of marcasitic pyrite."

As a paramorph consists simply of minute crystals of the one substance massed together in a form which characterises the other, it is obvious that the oxidation coefficient must correspond to that substance of which the specimen really consists, not to that which it imitates, and such a curve would indicate the proportions in the case of a mixed crystal. It would further enable us to follow the possible artificial transformation of one into the other, and would serve as an aid in synthetic work. In such work it is frequently difficult to obtain products sufficiently well characterised to admit of positive identification in the absence of optical properties and well developed crystallisation. Such a curve would render us independent of these.

It is not possible to calculate the curve for mixtures of pyrite and marcasite from the oxidation coefficients of the two minerals. The oxidation coefficient is influenced by the concentration of the ferric and ferrous salts and the sulphuric acid. With the pure mineral these vary in a perfectly constant manner in the case of each sulphide, but when each mineral is decomposing in the presence of the products formed by the other, nothing short of an elaborate investigation could give us the data for calculating the effect in any case. It is therefore necessary to construct the curve empirically, from data obtained from a sufficient number of artificial mixtures.

Conditions under which the Oxidation Coefficients of Mixtures may be Determined.

We have seen that the oxidation coefficients of pyrite and marcasite are independent of the relative amounts of solvent and solute, and of the degree of comminution, and are constant for a given temperature and concentration provided the reduction be complete. The case of a mixture of the two is more complicated. Here each component deprives the other of a portion of the ferric salt, this effect being greater the greater the surface, or, what is the same, the finer the powder. An artificial mixture of given composition will therefore show a varying oxidation coefficient depending on the relative degree of fineness of the two components. Moreover, since a given volume of the standard ferric solution decomposes about 2·26 times as much marcasite as pyrite, the ratio between the surfaces is constantly changing, and it is no longer a matter of indifference whether a large or a small amount of material be acted on by a given volume of solution.

If, however, the minerals be ground together, and if the grinding be tolerably thorough, then, considering their approximately equal hardness, it may be assumed that the ratio of the surfaces for a given mixture will remain practically constant, and if the value of p be obtained by using such an excess of the mixture that this ratio is not appreciably altered during the experiment, or if the same amount of mixture and of solution be used in every case, it becomes possible to obtain concordant values of p and to construct the desired curve. The assumption that the ratio of surfaces remains constant during grinding is not absolutely true, but practically it is, as the duplicate results presented below were each obtained from different samples, so that the slightly varying hardness of different specimens and the different degree of fineness to which they are ground introduce no greater errors than those which are inherent in the method itself. In each case the materials were broken up so as to pass a 20-mesh but to be retained by a 60-mesh sieve, were carefully weighed, mixed, and ground so as to avoid all loss, and 1 to 1·02 grms. of the mixture and 250 c.m.^{3} of the solution were used in each experiment.

(To be continued).

NOTICES OF BOOKS.

The Carbides of Hydrogen ("Les Carbures d'Hydrogène"), 1851-1901. Experimental Researches. By M. Berthelot, Senateur. Three volumes. Paris: Gautier-Villars. 1901. Pp. 1431.

The work now presented to the chemical world contains the results of the author's researches on the carbides of hydrogen, and principally on their synthesis from their elements, which synthesis is the pivot on which all other syntheses in organic chemistry turn.

The formation of acetylene, of ethylene, of formene, and of benzine, the four fundamental carbides, that of the pyrogenic carbides, and the general methods for the hydrogenation of the carbides and other organic compounds, have been the constant study of the author for half a century, his first work in this direction being undertaken in the year 1851, and the last being completed in 1901.

This great work is divided into three volumes; Volume I. is on "Acetylene; the Complete Synthesis of Carbides of Hydrogen," and comprises two books. In Book I. the author fully describes the synthesis of acetylene, formene, ethylene, benzine, and the polymeric carbides of acetylene. Book II. is devoted to the derivatives of acetylene; that is to say, the compounds resulting from its union with elements, such as nitrogen, hydrogen, oxygen, the halogens, and metals.

In the second volume M. Berthelot describes his experiments with the pyrogenic and some other carbides belonging to the propylic acid camphenic series. This volume is also divided into two books. Book III. deals with the pyrogenic carbides derived from acetylene and from the most simple carbides, submitted to the action of high temperatures. He also examines the action of heat on the mixed carbides, and the thermo-chemical relations between these different carbides. Book IV. is on propylene, its isomer trimethylene, and the allylic series, as well as the terebenthenic, camphenic, and terpilenic carbides.

In volume three the author gives his experiments on the general formation of the derivatives of the carbides of hydrogen. This volume is divided into three books. Book V. deals with the hydrogenation of the carbides and organic compounds generally; this hydrogenation is effected by a method of universal application founded on the use of hydriodic acid, a method discovered by the author in the year 1857, and further developed in 1868. Book VI. treats of the oxidation of the carbides of hydrogen and their transformation into aldehydes and acids by various methods.

Finally, in Book VII. we have the synthesis of the alcohols by means of the carbides of hydrogen, either by hydratation or by oxidation; a general and direct method is also described, destined to establish the alcoholic function of various principles, such as Borneo camphor and cholesterine, which had not yet been regarded as alcohols.

These three volumes, which represent a scientific lifetime, are of the greatest value not only to specialists, but to those whose tastes incline them to keep up with the march of scientific knowledge and the discovery of truth, and will remain a monument to the greatness and fame of its author.

Beiträge zur Chemischen Physiologie und Pathologie. Edited by Franz Hofmeister. Band I., 7-9 Heft. Braunschweig: Friedrich Viewig und Sohn. 1901.

The December number of these "Beiträge" includes amongst others a paper on the mutual action of the enzymes upon one another, by A. Wroblewski and two collaborators, in which is established experimentally the general conclusion that this action is very limited, as proved in the cases of pepsin, trypsin, diastase, and invertin; also an exhaustive report of investigations as to

the seat of the synthesis of sulphuric acid ethers in the animal body, which Drs. Embden and Glaessner believe they have proved to be principally the liver. Of more purely chemical interest are two shorter papers on the Aromatic Groups in the Albumen Molecule and Glue respectively.

Elektrische Verbrauchsmesser der Neuzeit. By JOHANNES ZACHARIUS. Halle-a-S.: Wilhelm Knapp. 1901.

ACCORDING to the author of this book, the theory of the construction of the electricity meter has received but little attention in recent years, and the paucity of literature on the question has led him to make this attempt to bring together all that is at present known of the subject. The book commences with an historical account of the development of such apparatus, and includes sections on the standardisation, reading, and choice of a meter. A considerable part of the book is devoted to a list of patents of electricity meters and allied apparatus from the year 1881 to 1900 inclusive, together with short abstracts of the specifications. The diagrams and illustrations throughout the book are particularly plentiful and good.

Die Pflanzen-Alkaloide. By JUL. WILH. BRÜHL, EDVARD HJELT, and OSSIAN ASCHAN. Braunschweig: Friedrich Viewig und Sohn.

THIS monograph on plant alkaloids forms a part of the eighth volume of the revised edition of Roscoe and Schorlemmer's "Complete Text-book of Chemistry," and deals with all known plant alkaloids and bases related to them. It is not too much to say that it is probably the most complete book on the subject extant, and will no doubt appeal to a wide circle of scientific readers. The subject is treated chiefly from an historical point of view, and the accounts of classical work on the constitution of many of the substances are most interesting and stimulative.

The authors display a perfectly unbiassed spirit in putting before their readers views which may still be regarded as disputed; as, for example, in the case of Merling's and Willstätter's excellent work on the constitutional formula of tropine. As far as possible, the alkaloids are grouped together as derivatives of pyrolidine, pyridine, quinoline, and isoquinoline, but a large number which cannot be identified as derivatives of any of these bases are classed as "alkaloids of unknown constitution." Under this heading are included only those whose empirical formulæ are fairly well established; they are arranged according to the botanical families of the plants from which they are obtained. An appendix on gluco-alkaloids, and some alkaloids, such as artarine and fumarine, which appear to stand alone, is added. Such a work as this cannot fail to have a favourable reception, and the hope of the authors expressed in the preface, that the biologist, physician and pharmacologist, as well as the chemist, may find it useful and instructive, will without doubt be fulfilled.

Remarks on Molybdenum Oxides.—Marcel Guichard.—The author refers to the work of M. Klason and M. Bailhache on the subject of the blue oxides of molybdenum. His own researches lead him to the conclusion that the plurality of the blue oxides has not been demonstrated. The constitution of these bodies is perfectly unknown, several different formulæ being possible to represent each. It seems natural to attribute, to the blue oxide, a formula containing a molybdyle or a molybdenyl group, on account of such a formula corresponding to about 68 per cent of the metal. The author now suggests a formula containing molybdic acid and molybdenum oxide, because the compound behaves under all circumstances during its reactions as a molybdate of molybdenum oxide.—*Comptes Rendus*, cxxxiv., No. 3.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxiv., No. 3, January 20, 1902.

Properties of Lime at its Fusing-point.—Henri Moissan.—When graphite is placed in contact with lime at its fusing-point it becomes surrounded with a layer of calcium carbide, $CaO+3C=CaC_2+CO$. If the melted lime is in excess and the action is prolonged the carbide becomes oxidised and calcium liberated—$CaC_2+2CaO=3Ca+2CO$. If silicon is treated in a similar manner it at once becomes transformed into silica, and this forms a basic silicate in presence of excess of lime. In the case of boron, borate of calcium is formed; but if the experiment is of very short duration the residual boron becomes surrounded with little black crystals of calcium boride, CaB_6. A number of metals when treated in this way, particularly when oxidation does not take place, are easily obtained in a crystalline state by simple vaporisation in a bath of melted lime. At this high temperature boiling platinum unites with calcium. Percentages of from 2·54 to 3·01 of lime have been found in the non-volatilised metal.

Critical Constants and Molecular Complexity of Certain Organic Compounds.—Ph. A. Guye and Ed. Mallet.—The authors make a series of measurements on the critical constants of anisol, phenetol, *m*-cresol, aniline, dimethyl-aniline, and dimethyl-*o*-toluidine, and from the numbers obtained they conclude (1) that the capillary ascensions of aniline indicate a slight polymerisation in the liquid phase, at low temperatures (considerably under boiling-point); (2) the capillary ascensions of meta-cresol show that at temperatures near its boiling-point this compound is still partially polymerised in the liquid phase. The authors extend their research to the case of the nitriles, and find that the numbers obtained confirm and generalise MM. Dutoit and Friderich's conclusion.

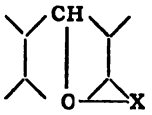
Certain Physical Properties of Selenium Hydride.—MM. de Forcrand and Fonzes-Diacon.—The authors determine the temperature of liquefaction and ebullition of selenium hydride, under atmospheric pressure; also its density in the liquid state at its boiling-point, the temperature of solidification, and its solubility in water; all experiments have to be effected as rapidly as possible, to avoid the spontaneous alteration of this gas in presence of mercury and under the influence of light. However, this decomposition is slower than up to the present time was believed; in darkness it is almost nothing at the end of two or three days, and the authors found only 15 per cent of free hydrogen in a specimen of pure selenium hydride, after a week of exposure to full daylight, in presence of mercury.

Decomposition of Acetylene during Combustion.—Fernand Gaud.—The author's experiments were at first performed on burning jets of chemically pure acetylene, then on jets of gas to which each of the ordinary chemical impurities was methodically added, and he shows that under certain circumstances the heat evolved by the substance of the jet can be transmitted to the gas and favours the decomposition into its elements, and it is certain that the presence of sulphuretted hydrogen, and of the thionic products, is sufficient to provoke this decomposition during the formation of the flame.

Action of Mono-halogenated Propionic Ethers on Sodium Acetylacetone.—Fr. March.—Resuming his research on the action of the halogenated ethers of saturated acids of the fatty series on sodium acetylacetone, the author causes α-bromopropionate and β-chloropropionate of ethyl to react on this sodium salt. These

two reactions enable him to prepare α-methyl-ββ-diacetyl-propionate of ethyl and ββ-diacetylbutyrate of ethyl.

Tribromo- and Triiodo-dinaphthoxanthonium; also the Bibromated Hydrobromic Ethers and Biodated Hydriodic Ethers of the supposed Binaphthyleneglycol.—R. Fosse.—In a preceding paper the author attributed the constitutional formula—



to the mono-halogenated derivatives to the xanthenes, hbtained either by the action of the hydracids on the oydrols, or by the action of the halogens on the xanthenes. The bodies represented by this formula, where the oxygen behaves as a tetrad, by uniting one liaison to the opposed carbon group, ought to be able to fix two atoms of a halogen giving tri-halogenated derivatives. Experiment verifies this hypothesis.

Chemical Modifications taking place in a Plant subjected to the Influence of Sodium Chloride.—E. Charabot and A. Hébert.—The authors' investigations lead them to the conclusions that the addition of sodium chloride to the soil in which a particular plant is growing causes (1) an increase in the percentage proportion of organic matter in the plant; (2) a relative loss of water. At the same time that it exerts this double influence on vegetable life, sodium chloride favours etherification, and retards the transformation of menthol into methone.

MISCELLANEOUS.

Royal Institution.—A General Monthly Meeting of the Members of the Royal Institution was held on the 3rd inst., Sir James Crichton-Browne, M.D., F.R.S., Treasurer and Vice-President, in the Chair. Lord Iveagh, William Asch, Esq., Rev. S. J. Norman, Hunter Finlay Tod, Esq., James Martin White, Esq. George Westinghouse, and Lieut.-Col. Richard de Villamil were elected Members. The thanks of the Members were returned to Sir Frederick Bramwell, Bart., F.R.S., for his donation of £100, and to Mr. Frank McClean, F.R.S., for his donation of £50 to the Fund for the Promotion of Experimental Research at Low Temperatures. It was announced that the following valuable relics of Michael Faraday, bequeated to the Royal Institution of Great Britain by the late Mr. Thomas J. F. Deacon, of Newcastle-on-Tyne, had been received:—Medals of Silver and Bronze (numbering twenty in all), and including the Fuller Medal of 1828, two Copley Medals of 1832 and 1838, two Newton Medals of the Royal Society, 1833 and 1838, and the Rumford Medal of 1846; and two Foreign Orders contained in a small mahogany box. A Book of Portraits and Autographs, including original letters from the Prince of Wales and Prince Alfred (written in 1856), Louis Napoleon, Emperor of France, Humphry Davy, Thomas Young, Humboldt, John Dalton, Whewell, Mary Somerville, and many others. A Daguerrotype of a consultation of Faraday with Professor Daniell; a Drawing in colours of the Laboratory of the Royal Institution, by a niece of Sir John Moore; and a Manuscript Book entitled "A Class Book for the Reception of Mental Exercises, instituted July, 1818," containing contributions by Faraday. The late Mr. Deacon requested that the Medals and Orders should be preserved with an inscription showing that Margery Ann Reid and Caroline Deacon (*née* Reid), nieces of Faraday's wife, often lived with Faraday and his wife during the most brilliant period of his life, and are mentioned in Dr. Bence Jones's "Life of Faraday."

MEETINGS FOR THE WEEK.

MONDAY, 17th.—Society of Arts, 8. (Cantor Lectures). "Personal Jewellery from Prehistoric Times," by Cyril Davenport.

TUESDAY, 18th.—Royal Institution, 3. "The Cell—its means of Offence and Defence—Immunity," by Allen Macfadyen, M.D., B.Sc.

— Society of Arts, 4.30. "The French-Canadian Relationship to the Crown," by W. T. R. Preston.

WEDNESDAY, 19th.—Society of Arts, 8. "The Use of Balloons in War," by Eric H. Stuart Bruce, M A.

— Chemical, 5.30. Ballot for the election of Fellows. "Enzyme Action," by A. J. Brown. "On the Velocity of Hydrolysis of Starch by Diastase, with some remarks on Enzyme Action," by H. T. Brown and T. A. Glendinning. "Polymerisation Products from Diazoacetic Ester," by O. Silberrad. "Condensation of Phenols with Esters of Unsaturated Acids" (Part VII.), by S. Ruhemann and H. E. Stapleton. "The Union of Hydrogen and Oxygen," by H. B. Baker.

— Microscopical, 8. "Polarising with the Microscope," by E. M. Nelson.

THURSDAY, 20th.—Royal Institution, 3. "The Scot of the Eighteenth Century—III., With his Books," by the Rev. John Watson, D.D. (Ian Maclaren).

FRIDAY, 21st.—Royal Institution, 9. "Musical and Talking Electric Arcs," by W. Duddell.

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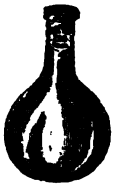

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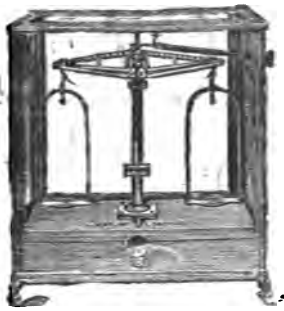

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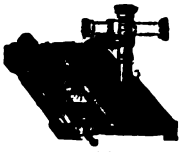

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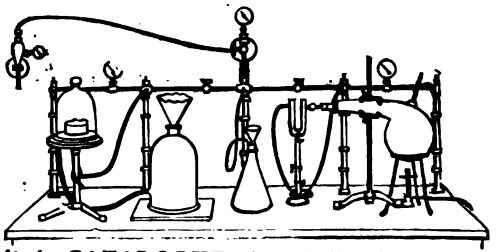

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VOL. LXXXV., No. 2204

THE STRATIFICATIONS OF HYDROGEN.*

By Sir WILLIAM CROOKES, F.R.S.

THE following pages give the outcome of attempts to prepare pure hydrogen, and experiments on the stratifications exhibited by the purified gas under the influence of an induction current. The researches were commenced in 1884, and have been continued intermittently to the present time.

The original apparatus consisted of a vacuum tube of soda-glass, 6 inches long and ¼ inch wide, having sealed-in aluminium terminals at each end. The pole at one end was ring-shaped, at the other pointed. The vacuum tube was connected to the mercury pump at one end, a tightly packed phosphoric anhydride tube intervening. At the other end was another phosphoric anhydride tube, a hydrogen generator of zinc and dilute sulphuric acid, and a tap to control the flow of gas. The hydrogen was passed through the apparatus for some hours, and the whole was exhausted to a high point, re-filled, and again exhausted. This was repeated many times; but, on exhausting to the stratification-point, I could get no spectrum which did not show, in addition to hydrogen, also mercury.

The apparatus was therefore modified. Strips of palladium foil were charged with hydrogen by the electrolysis of dilute sulphuric acid; a 4-cell Grove's battery being used for one hour. After drying, the palladium strips were put in a glass tube, and sealed between the generator and vacuum tube. At first, crude gas from the generator was used to wash out the apparatus, and after many fillings and exhaustions—the last to the highest possible point—the generator and tap were sealed off, leaving only the palladium and drying tubes attached to the apparatus. A portion of the palladium was now gently heated; the gauge sank 12 c.m., when it was again well exhausted and a little more hydrogen liberated. This was repeated three times, when the tube was exhausted to the stratification-point—about 4 m.m.

Parti-coloured Stratifications.

The strata were twelve in number, and of a slightly concavo-convex button-shape, each of a blue colour on the convex side facing the negative pole, and pink on the other side. On reversing the current, the buttons faced round, always presenting the blue face to the negative pole. Examination with a spectroscope showed strong hydrogen lines in the pink parts, and both hydrogen and mercury in the blue parts. Fig. 1 shows the appearance at this stage.

The exhaustion was now raised to 2 m.m., when the whole of the blue faces of the parti coloured buttons suddenly migrated to one bright blue, well-formed button, nearest the negative pole all the other buttons remaining pink. The appearance is shown in Fig. 2. Round the negative pole an indistinct halo showed both mercury and hydrogen; but on the blue button mercury only was detected, not a trace of even the brightest hydrogen line being there seen. On the pink portions the hydrogen lines were in excess; but mercury could be seen all along the tube.*

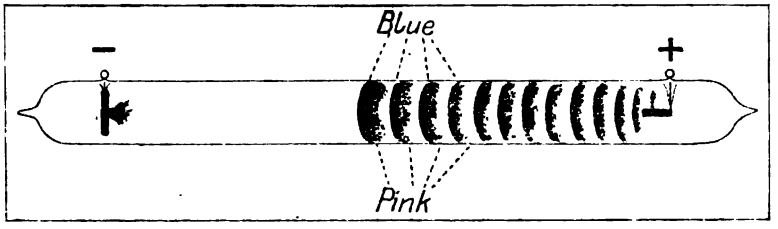

FIG. 1.

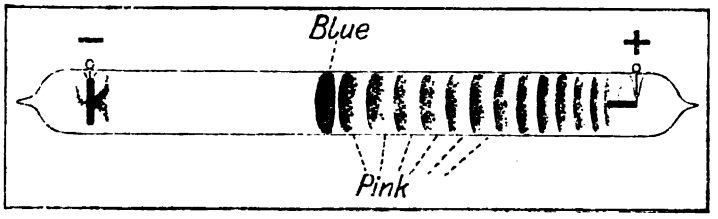

FIG. 2.

* A Paper read before the Royal Society, February 6, 1902.

* I have been unable to find any reference to this concentration of the blue constituents of the strata into one single button at the end near the negative pole. In the classical researches of Messrs. De la Rue and H. W. Müller on the "Electric Discharge with the Chloride of Silver Battery," numerous references are made to the blue and pink parti-coloured character of the strata, and also to the change of colour from all blue to all pink which follows a change in the electrical conditions. Thus we read:—"Thirty-nine strata, the convex side being blue and the broader concave side reddish." "Twenty-one strata, very blue on the convex face, pink on the concave."—*Phil. Trans.*, vol. clxix., p. 175.

Sometimes the stratifications are described as all of one colour:—"Twenty-one very blue strata." "The strata were blue, and sixty-one in number." "Showing twenty-one double strata, intensely blue, but with a carmine line between the components."—*Phil. Trans.*, vol. clxix., pp. 175, 190.,

Frequently a change from all of one colour to all of the other colour is recorded. Thus:—"The tube was filled to within 1 inch of the negative with strata; all these were blue, but they turned pink when 200,000 ohms resistance was introduced. When 7,590,000 ohms resistance was introduced, a very close and somewhat agitated pink stratification was produced." "Thirty-four steady blue strata were

At 1 m.m. pressure very little hydrogen could be detected by the spectroscope, the stratifications had almost disappeared, and mercury was strong throughout the tube.

Elimination of Mercury Vapour.

The mercury apparently diffused into the tube from the pump and many devices were adopted to keep it out. Long glass tubes filled with purified sulphur broken into coarse pieces were partially successful, but something (probably sulphur) was communicated to the hydrogen which interfered with the purity of the colours. Also the sulphur did not prevent a little mercury diffusing in if the tubes were left on the pump all night.

Experience showed that mercury was very difficult to eliminate from a tube once it had gained access. It adheres to the walls of the tube, and defies detection in the cold, but becomes visible in the spectroscope as soon as the tube is heated by a lamp. Therefore a new hydrogen tube was made, and connected with tubes containing sulphur, bright copper turnings, and phosphoric anhydride; the copper intervening to keep out the sulphur. Here, again, mercury was detected after some days, but only in minute traces. In each of these cases occurred the phenomena of the pink and blue strata at one exhaustion, changing to all-pink with a concentration of the blue to the front on increasing the vacuum; the single blue button showing the mercury spectrum. Continued pumping diminished the hydrogen, but apparently did not affect the mercury to the same extent. It appears that the great diffusibility of the hydrogen causes it to be readily pumped out, whilst the mercury is continually being replenished by diffusion from the pump.

A slight difference is produced in the purity of the colours of the strata according as aluminium or platinum poles are used. A pair of vacuum tubes was made, one having the usual shaped aluminium poles, the other having platinum poles of a special construction. Each terminal was of double wire, at one terminal bent into the form of a ring, and at the other a straight pole. The ends of the wires forming the poles were sealed through the tube close together but not touching, and terminated in loops outside, so that they could be raised to red or white heat by connecting them with a few battery cells. The arrangement will be readily understood by reference to the accompanying drawings (Fig. 3). Thus heat could easily

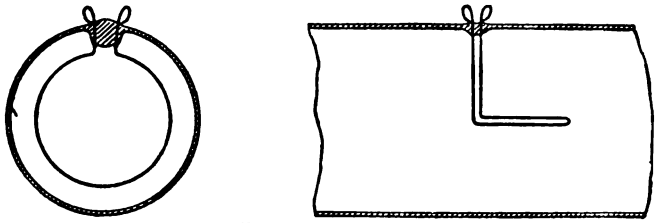

FIG. 3. (Full size).

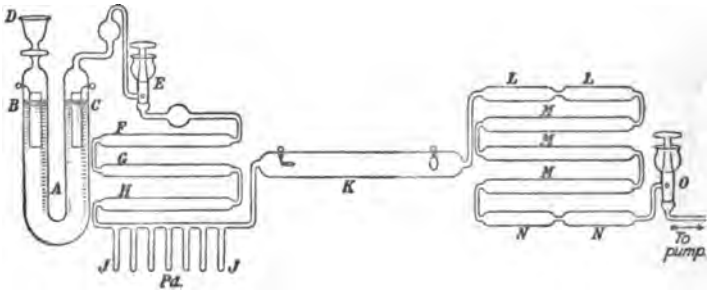

FIG. 4.

be applied during exhaustion, first to one pole and then to the other, even while the induction spark was passing. At first much gas was liberated from the platinum, but by repeated heating, pumping, and passing the spark, all the occluded gas was abstracted, and then the fillings with hydrogen and subsequent operations were commenced.

The general plan of the apparatus is shown in the drawing (Fig. 4). At the end furthest from the pump is the hydrogen generator, A, consisting of a U-shaped tube filled with dilute sulphuric acid, having in one leg a plate of amalgamated zinc, B, and in the other a sheet of platinum, C. Both the platinum and the zinc are connected metallically to platinum wires sealed through the glass. A funnel with a stopper, D, sealed to the outer limb of the generator admits dilute acid when required. A tap, E, on the other limb enables the reservoir of hydrogen to be disconnected from the rest of the apparatus. Following this tap is a battery of three tubes, one F, containing small lumps of dry caustic potash; the second, G, and the third, H, tubes containing phosphoric anhydride. Between the second phosphoric anhydride and the vacuum tube is another tube having sealed on to it, comb-like, seven projecting arms, J J, each containing a strip of palladium foil saturated with hydrogen.

The vacuum tube, K, is 8 inches between the terminals

produced; on introducing 200,000 ohms resistance, the strata turned pink." "With 3600 cells the strata were blue and sixty-one in number. With 700,000 ohms resistance the strata were reduced to eighteen and turned pink." "A change of current frequently produces an entire change in the colour of the strata. For example, in a hydrogen tube, from a cobalt-blue to a pink."—*Phil. Trans.*, vol. clxix., pp. 183, 190, 231.

Very few spectrum observations were made by the authors, and they are not very definite in character. One tube, which gave ten luminosities, was examined with the spectroscope. "The C and F lines were brilliantly seen in the glow around the negative terminal, but were not visible in the spectrum of the nebulosities, notwithstanding that they were brighter than the negative glow; there were blue, green, and red visible, but not the characteristic green and red lines of hydrogen." In another tube, "The characteristic hydrogen lines were very brilliant when the spectroscope was directed to the glow around the negative terminal, but quite a different spectrum was seen on a bright stratum, with mercury lines in the orange." "The hydrogen lines could not be seen either in the strata or the glow on the negative ring, but instead of them mercury lines came out strongly. There is reason to think that at this stage there was little gas except mercury vapour in the tube." "A change of current frequently . . . changes the spectrum of the strata; moreover, the spectra of the illuminated terminals and the strata differ."—*Phil. Trans.*, vol. clxix., pp. 180, 216, 232.

and ⅜ inch diameter; it comes next to the comb, and then between it and the pump is a battery of tubes, each 12 inches long, to keep out the mercury. The first tube, L, is divided by a constriction in the middle, and contains, in the half next the vacuum tube, bright metallic copper, in the other half sulphur. The three next tubes, M, M, M, contain sulphur, but in the middle of each are placed a few grains of iodine separated from contact with the sulphur by a plug of asbestos on each side. The sulphur is prepared by keeping it fused at a temperature a little below its boiling-point till bubbles cease to come off, so as to get rid of water and hydrogen compounds. It is then allowed to cool, and is pounded and sifted so as to get it in the form of granules, averaging a m.m. in diameter. Ignited asbestos is packed at each end of the tubes to keep the contents from blowing out when the vacuum is proceeding, or air is suddenly let in.* Next follows a tube, N, N, constricted in the middle, containing in the first half phosphoric anhydride, and in the second finely powdered dry caustic potash. A tap, O, connects the apparatus with the pump to prevent diffusion of mercury when the pump is not in use. All parts of the apparatus were built up in place and sealed together with the blowpipe. The glass was new, and the apparatus had been kept apart from mercury until it was sealed together.

The apparatus was exhausted from air, the tap E being closed and D open. Electrolysis was then commenced (D being closed), and the tap E was slightly turned until the escape of hydrogen into the apparatus was equal to the speed of its generation. The apparatus was filled, and several times exhausted, until no improvement in the spectrum or stratifications could be seen. The electrolytic cell was then sealed off at a narrow constriction between the first potash tube, F, and the phosphoric anhydride tube, G. After good exhaustion one of the branch tubes of palladium was heated, when the gauge sank several centimetres. Exhaustion and re-filling from fresh palladium were repeated until no alteration was detected in the appearance of the strata. Then, for the first time, I obtained hydrogen strata showing no blue, either throughout the tube or concentrated in front, whilst the most careful examination showed no mercury. The stratifications were all pink, and showed the hydrogen lines strongly.

Many disadvantages were noticed in the apparatus just described, the chief being the danger of introducing more impurities than were kept out by the copper, sulphur, and iodine tubes. The palladium method of introducing hydrogen was not altogether satisfactory, as only small quantities could be dealt with, and occasionally at a critical point the store was exhausted. Also, the electrolytic generator of hydrogen was too small. It was decided, therefore, to devise and fit up an entirely new piece of apparatus. In this another method was used for keeping out the mercury. It had been noticed that the diffusion of mercury from the pump proceeded the more slowly as the distance from the pump and the narrowness of the connecting tubes increased. It was thought that by introducing a long narrow spiral between the pump and the apparatus, one complicated system of tubes, with their attendant dangers, could be removed; the result showed this supposition to be correct. Two vacuum tubes were employed, one having aluminium the other platinum terminals. The hydrogen generators were increased in size and number, and were so distributed that they could be sealed off one after [illegible]ther during the progress of the experiment.

(To be continued).

* An apparatus of this kind was briefly described by the author in a paper read before the Royal Society in 1885 ("Radiant Matter Spectroscopy—Part II., Samarium," *Phil. Trans.*, vol. clxxvi., p. 693, June 18, 1885). The apparatus was used to prevent mercury from getting into the small radiant-matter tubes employed in the research.

A BIBLIOGRAPHY OF STEEL WORKS ANALYSIS.

PART XI.

By HARRY BREARLEY.

(Continued from p. 79).

COPPER.

III. VOLUMETRIC ESTIMATIONS.

a. Cyanide Process.—

1561. FIELD (*C. N.*, i., 25, &c.).—Not necessary to filter off precipitated $Fe_2(HO)_6$. Arsenic easily separated from KCN solution. Hg, Ag, Zn, Ni, Co, and Mn interfere. Describes also the following methods: — Titration with alkaline sulphide (Pelouze); Reduction to Cu_2O and titration with permanganate (Treille); precipitation with sugar and titration with permanganate (Mohr); titration with Fe_2Cl_6 and $KMnO_4$ (Schwarz); precipitation of Cu with Fe and estimation in various ways; titration with KI and $K_2Cr_2O_7$ (Streng); estimating metal used in reducing CuO to Cu_2O; the iodide process; colour comparison of AmHO solutions; weighing as Cu_2S+CuO; and the sulphocyanide process (Rivot).

1562. FLECK (*C. N.*, ii., 35).—Results vary with excess of AmHO; uses Am_2CO_3 instead, and an indicator for end-reaction.

1563. STEINBECK (*C. N.*, xix., 207).—Precipitate Cu from solution of ore with Zn and Pt, dissolve in HNO_3, and titrate. Influence of Pb, Zn, AmHO compounds and heat observed.

1564. THOMSON (*C. N.*, xxxiii., 152).—Observes influence of many elements on titration; that of Zn, Cd, Mn, Ni, Sn, Hg, Ag, Au, Pt, and Pd most pronounced. AmHO and its salts cause variations.

1565. ULBRICHT (*C. N.*, xliv., 82).—Sample to be treated exactly as standard, and to be as nearly as possible identical.

1566. BERINGER (*C. N.*, xlviii., 111).—Results influenced by mode of operation and temperature. AmHO exerts same influence whether as sulphate, nitrate, or chloride. Soda salts no influence, except carbonate, sulphite, and nitrite. Determines conditions under which proportional results are obtained.

1567. DAVIES (*C. N.*, lviii., 131).—Results are more concordant when the solution is made alkaline with Na_2CO_3 instead of AmHO.

1568. FESSENDEN (*C. N.*, lxi., 183 and 253).—Confirms 1567; the end-reaction is more decided.

1569. BREARLEY (*C. N.*, lxxvi., 189).—The AgI indicator (580) is applied to the KCN estimation. The results are better, particularly when the solution is made alkaline with Na_2CO_3.

1570. BREARLEY (*C. N.*, lxxvi., 291, 303).—Influence of many elements on 1569 process observed. Interference of Zn, Mn, and Cd avoided; Cr_2O_3 salts injurious. Organic acids and pyrophosphate used to keep precipitable bodies in solution.

1571. DONATH and TELLER (*C. N.*, lvii., 102).—Ore ignited with Zn powder, boiled with H_2SO_4, Cu dissolved from associated gangue with HNO_3, and titrated with KCN.

1572. JOHNSON (*J. S. C. I.*, 1889, 603).—Cu precipitated from solution of pyrites with KCNS and $SnCl_2$, dissolved in nitrohydrochloric, and titrated with KCN; or the CuCNS dissolved in $Fe_2(SO_4)_3$ and resulting FeO titrated with $KMnO_4$. $SnCl_2$ is better than any other reducer.

1573. ELLIS (*J. S. C. I.*, 1889, 686).—Tabulated results showing influence of zinc. The solution may stand an indefinite time after adding AmHO without spoiling.

1574. CLENNELL (*J. S. C. I.*, 1900, 14).—Cu in KCN solutions estimated by adding H_2SO_4 until a precipi-

tate of $Cu(CN)_2$ appears, then an excess of H_2SO_4, and finally titrating the free acid in a fraction of the filtrate with Na_2CO_3. (See also 1580).

b. Iodide Process.—

1575. Westmoreland (*J. S. C. I.*, 1886, 48).—Analysis of ores, pyrites, &c. Neither Ag, Zn, Cd, As, Bi, Mn, or Sn interfere; nor does Fe materially in the presence of Na_3PO_4 or As_2O_3. Also critical account of the electrolytic and dry assay processes and the assay of Au in bar Cu.

1576. Westmoreland (*C. N.*, lviii, 76, 108, and 132).—Shows that the iodide process is superior to the electrolytic for all kinds of Cu compounds.

1577. Williams (*C. N.*, lviii., 272).—Pb and As exert a prejudicial effect on the titration.

1578. Haupt (*C. N.*, lxx., 206).—Titration not interfered with by Zn or Pb; it is by Ni and Fe. Cu precipitated from Fe solutions with Zn. KI detects one Cu in 500,000. Experiments on the ignition of Cu_2S.

1579. Bruyn and Van Leent (*J. C. S.*, lxii., 753).—Operate in stoppered flasks. Ni, Mn, and Fe as sulphates and free acid no not interfere; Bi does.

1580. Dulin (*C. N.*, lxxii., 70).—Describes KCN method and its limitations. KI process influenced by large amounts of alkaline salts and Bi. For either titration first precipitate Cu with Al. Electro-deposition from solutions containing much HNO_3.

1581. Low (*C. N.*, lxxiv., 52).—First precipitate Cu with Al; then neither Ag, Pb, Bi, or As interfere. Procedure for ores.

1582. Willenz (*C. N.*, lxxvi., 243).—KI process applied to estimation of Cu in pyrites.

1583. Shelby (*J. S. C. I.*, 1900, 776).—FeO formed by hypo. and then sheet Zn added. Nascent H and the previously liberated S form H_2S, which precipitates the Cu. Estimation with KI or KCN after dissolving in HNO_3, &c. (See also 1591).

c. Various Volumetric Processes (see also 1572).—

1584. Fleischer (*C. N.*, xix., 206).—Precipitate as subiodide with KI and $SnCl_2$, dissolve precipitate in $Fe_2(SO_4)_3$, and titrate FeO with $KMnO_4$. Or, precipitate Cu with KCNS and SO_2, digest CuCNS with NaHO, and estimate Cu_2O as before. Pb, Ag, and Hg are the only interfering metals, and these are separated with H_2SO_4, HCl, and $SnCl_2$ respectively.

1585. Henriques (*J. C. S.*, lxiv., ii., 345).—Precipitate with SO_2 and standard KCNS. Estimate excess KCNS with $AgNO_3$ and $Fe_2(SO_4)_3$ indicator.

1586. Meade (*C. N.*, lxxx., 67).—Decompose precipitated CuCNS with KHO, add collected $Cu_2(OH)_2$ to acid $Fe_2(SO_4)_3$ and titrate FeO with $KMnO_4$.

1587. Parr (*J. S. C. I.*, 1900, 1148).—Decompose CuCNS with NaHO, destroy $Cu_2(OH)_2$ with $KMnO_4$ in alkaline liquid, then acidify and titrate KCNS with $KMnO_4$. 1 c.c. N/10 $KMnO_4$ = 0·0009 Cu.

1588. Caven and Hill (*J. S. C. I.*, 1897, 981).—Add Cu_2O to excess $KMnO_4$ in H_2SO_4 and titrate unreduced portion with oxalic acid.

1589. Dederichs (*J. C. S.*, lxxvi, ii., 813).—Digest HNO_3 solution of brass with $NaHSO_3$, add standard AmCNS, and titrate excess with $AgNO_3$ in aliquot filtrate.

1590. Zecchini (*J. S. C. I.*, 1899, 710).—Add excess of solution containing hypo. and AmCNS and titrate with iodine. Neither AmCNS nor CuCNS react with iodine.

1591. Garrigues (*C. N.*, lxxvii., 41 and 52).—End-point of iodide process sharper in H_2SO_4 than in $C_2H_4O_2$ solution. Decompose precipitated CuCNS with standard NaHO and titrate excess with acid and methyl-orange. Conjoint presence of Zn and Sb interfere. Some important points in the analysis of bearing-metals, bronzes, &c.

1592. Weil (*C. N.*, xxiii., 49; and xlvi., 284).—HCl solution of Cu titrated at boiling-point with $SnCl_2$ to decolorisation. $HgCl_2$ may be used as indicator. Fe and Ni interfere.

1593. Jean (*J. C. S.*, lxiv., ii., 493).—Estimation of Cu, Fe, Sb, and Zn in minerals by Weil's process.

1594. Kunsel (*C. N.*, viii., 38).—Titrate hot AmHO solution with Na_2S, using freshly precipitated ZnS as indicator. Ni or Zn do not interfere. Jean (*C. N.*, lxxvii., 172) uses $PbCO_3$ paper as indicator; the process separates As. Borntrüger (*J. C. S.*, lxvi., ii., 120) uses a ferrocyanide indicator, and says Cu-Zn alloys may be thus titrated.

1595. Casamajor (*C. N.*, xlv., 167).—Titration of AmHO solutions as in 1594 until blue colour goes is inaccurate. Precipitates instead from alkaline tartrate solutions until no further cloud forms. Same process for Pb, &c.

1596. Balling (*C. N.*, xlvii., 248).—The sulphide of Cu (Pb, Zn, or Hg) is digested with $AgNO_3$ and excess Ag titrated with KCNS.

1597. Luckow (*J. C. S.*, lxiv., ii., 242).—Titration of Cu-Sn-Sb alloys with ferrocyanide. Ranter (*J. C. S.*, lxx., i., 3).—The precipitate always retains $K_4Fe(CN)_6$. Spica (*J. C. S.*, lxx., ii., 127) uses Fe_2Cl_6 indicator.

1598. Quessand (*J. C. S.*, xlviii., 441).—Precipitate with slight excess of $K_4Fe(CN)_6$, and then measure the alkaline solution of Rochelle salt needed to destroy the precipitate.

1599. Donath and Hattensaur (*J. S. C. I.*, 1890, 554).—Cu precipitated with ferrocyanide from alkaline tartrate solutions. Fe should be present, so that end-reaction (blue) may be noted by adding acetic acid to test drop.

1600. Bernthsen (*C. N.*, xliii., 79).—Titrate cupric solution with hypo. in an atmosphere of H. When colour is nearly gone add indigo to heighten end reaction.

1601. Lagrange (*C. N.*, xxx., 241).—Precipitate with KHO, convert to tartrate, and reduce to Cu_2O with standard glucose.

1602. Muller (*C. N.*, xxxi., 143).—Measure volume of H needed to reduce CuO to Cu in a closed tube.

1603. Vitali (*J. C. S.*, lxviii., ii., 140).—Mix $CuSO_4$ with KI and starch, add H_2SO_3 to decolorise, and estimate liberated H_2SO_4 with standard alkali.

1604. Etard and Lebeau (*C. N.*, lxi., 137).—Add hydrobromic acid to concentrated solution, and then standard $SnCl_2$ until the violet colouration is discharged.

1605. Purgotti (*J. C. S.*, lxxii., ii., 349).—Boil solution with excess NaCl and hydrazine sulphate. The evolved N is measured at normal temperature and pressure.

1606. Lescœur (*J. C. S.*, lxxiv., ii., 484).—Cu is precipitated as oxide, dissolved in excess acid, and titrated back with alkali. Cu_2O obtained with glucose titrated simultaneously. See also Ruoss (*J. C. S.*, lxxiv., ii., 644).

IV. Colorimetric Estimations.

1607. Carnelley (*C. N.*, xxxii., 308).—The colour formed with ferrocyanide in neutral solution is compared with standards. $AmNO_3$, &c., heighten the colour. Many alkaline and earthy salts do not interfere. Detects (so does H_2S) 1 part in 1,500,000. See also Iles (*C. N.*, xxxiv., 16) and Blunt (*C. N.*, xxxiii., 7).

1608. Lucas (*C. N.*, lxxix., 67).—Process 1607 used for estimating O in Cu and Cu in Fe. Separation of

Fe and Cu not complete after several re-precipitations.

1609. Muir (*C. N.*, xxxiii., 11).—Add H_2S and compare with standards. Determines minimum and maximum which can be estimated thus.

1610. Heath (*C. N.*, lxxvi. 184).—Comparison of AmHO solutions. Permanent standards prepared from $CuSO_4$. See 1538.

V. Detection.

1611. Bellamy (*C. N.*, xx., 228).—An alcoholic tincture of campeachy wood detects 1 part Cu (or Fe) in 20,000,000 parts of water.

1612. Wagner (*C. N.*, xlv., 35).—Limits of following tests are:—Ferrocyanide, 1 in 200,000; AmHO, 1 in 25,600; xanthogenate, 1 in 900,000.

1613. Aliamet (*C. N.*, lvi., 160).—Pyrogallic acid in saturated Na_2SO_3 gives red colour with Cu. (*C. N.*, lviii., 293).—Test by no means characteristic.

1614. Cailletet (*C. N.*, xli., 160).—Add pyrogallic acid in ether to the oil; a brown colour = Cu.

1615. Anon. (*C. N.*, xliii., 117).—One one-hundredth m.grm. Cu can be detected by the HBr test.

1616. Deniges (*C. N.*, lix., 168; and *J. C. S.*, lxviii., ii., 330).—A more stable form of reagent for above test is saturated KBr in H_2SO_4.

1617. Sabatier (*J. S. C. I.*, 1894, 1092).—KBr and H_3PO_4 may be used instead of HBr. Detects one-tenth m.grm. Cu.

1618. Kern (*C. N.*, xxxiv., 78).—Precipitate acid solution of iron with AmHO, acidify evaporated filtrate, and add Mg. Traces of precipitated Cu observed with the microscope.

1619. Woodcock (*C. N.*, xxxvi., 240).—Precipitate Cu with Zn-Pt element; expose Pt to Br+HBr; the violet colour detects 0·001 m.grm. Cu.

1620. Jaworowsky (*J. C. S.*, lxxii., ii., 285).—Shake AmHO solution with phenol; with traces of Cu, solution becomes turbid and blue.

1621. Bach (*J. S. C. I.*, 1899, 401).—Mixture of formaldehyde and hydroxylamine hydrochloride gives violet colour with one part Cu in a million.

1622. Cazeneuve (*J. C. S.*, lxxviii., ii., 627).—Diphenylcarbazide in benzene gives a violet colour when shaken with a cupric solution.

1623. Lenoble (*J. C. S.*, lxiv., 554).—Potassium mercuric iodide throws down bright red precipitate from cupric solutions.

1624. Thoms (*C. N.*, lxiv., 190).—Depends on yellow colour formed with KI. One part in 200,000. See 1578.

1625. Gerland (*C. N.*, ix., 73) and Chapman (*C. N.*, xii., 231).—Blowpipe tests for Cu.

1626. Schiff (*C. N.*, i., 191) and Orlowski (*C. N.*, xlvii., 24).—Tests for cuprous oxide.

VI. Miscellaneous.

1627. Thomsen (*C. N.*, xxxix., 185).—Precipitate formed with H_2S is not CuS, but a mixture of S and a lower sulphide. See also Coppock (*C. N.*, lxxiii., 262; and lxxvi., 231) and Brauner (*C. N.*, lxxiv., 99).

1628. Anon. (*C. N.*, xlvi., 120).—$Cu(NO_3)_2$ mixed with $Pb\bar{A}_2$ is dissolved by NaHO and not re-precipitated on boiling.

1629. Debray (*C. N.*, xlvii., 304).—CuS is dissolved in considerable quantities by Am_2S if Mo is also present.

The following refer to papers which deal generally with the estimation of the impurities of commercial copper:—

1630. Abel and Field (*C. N.*, iv., 264); Abel (*C. N.*, ix., 124); Fresenius (*C. N.*, xlvii., 18); Kuhn (*J. S. C. I.*, 1884, 374); Jean (*J. S. C. I.*, 1896, 677); Hampe (*J. S. C. I.*, 1894, 421; and 1897, 1043); Keller (*J. C. S.*, lxviii., ii., 186); Warren (*C. N.*, lv., 62; and lxxiii., 37); Benneville (*J. C. S.*, lxvi., ii., 297 and 298); Murman lxxii., ii., 346); Hollard (*J. S. C. I.*, 1897, 166 and 763; and *C. N.*, lxxxi., 258), electrolytic; Clark (*J. S. C. I.*, 1900, 27); Ulke (*J. S. C. I.*, 1900, 170).

The following deal with the estimation of As in Cu:—

1631. Clarke (*J. S. C. I.*, 1887, 353); Platten (*J. S. C. I.*, 1894, 324); Pattinson (*C. N.*, xlv., 136); Heath (*J. S. C. I.*, 1897, 699).

The following deal with the estimation of O in Cu:—

1632. Aubel (*C. N.*, xix., 238); Sabatier (*J. C. S.*, lxxii., ii., 261); Anon. (*C. N.*, xxxvi., 241); Lucas (*C. N.*, lxxx., 86); Dewey (*J. S. C. I.*, 1889, 134); Blount (*J. S. C. I.*, 1896, 296); Archbutt (*J. S. C. I.*, 1900, 1148). The estimation of O is also dealt with in many papers under 1630.

1633. Whitehead (*J. S. C. I.*, 1895, 513).—Fe in Cu.

1634. Smith *C. N.*, lxxii., 76).—Au and Ag in copper. See also 1575.

(end of part xi.).

ON RADIO-ACTIVE LEAD.

By F. GIESEL.

From a large quantity of the mother-liquor of barium-radium bromide resulting from the technical working of about 2000 kilogrms. uranium ore, I had separated (by means of ammonia and consequent purifying with sulphuric acid) about 3 m.grms. of an intensely radio-active substance which behaved like lead, and resembled preparations of radium in its action (compared on the phosphorescent screen). My interest in the substance increased when I found unexpectedly, after a year had elapsed, that it still exhibited strong radiation. I therefore gladly welcomed Demarçay's proposal to undertake an exact spectral examination.

As the small residue of sulphide (about 1 m.grm.) could not easily be separated mechanically from the small filter, the latter was ignited (with the substance), and evaporated with concentrated hydrochloric acid. The resulting compound, which consisted of crystals resembling lead chloride, was sent to M. Demarçay.

I received from him on December 11th, 1901, the following communication:—

"Your substance influences the platinocyanide screen.

"The spectrum of the hydrochloric acid solution was principally that of lead, which must form the bulk of the substance.

"There are traces present of calcium, barium, strontium, chromium, aluminium, manganese, bismuth, tin, slight traces probably of molybdenum, also of yttrium perhaps arising from my laboratory; magnesium in marked degree; iron to a remarkable extent.

"These substances represent the sum total of the lines of known origin, the strong, predominating, weak, and very weak, with the exception of two lines, also very weak, but not to be passed over. They cannot be identified amongst those of known origin.

"One of the lines, λ 3659·6, occurs with an extremely weak iron line. It also coincides with a line of the air spectrum, which is very weak.

"I do not believe that this line belongs either to the iron or the air, or to both; without, however, wishing to assert this positively.

"The second extremely weak line, λ 4116·8, which stands next to a strong line of platinum, cannot be identified with any other.

"I attach a certain importance to these two lines, particularly on account of the unexpected absence of radium. Its lines are absolutely wanting. If it is pre-

sent it must be so in very small quantity. It may possibly be that the radio-activity of your substance arises from a hypothetical body, which gives these two lines and some of the infinite number of weak ones, which have not been mentioned."

This examination admits of another explanation of the radio-activity of my substance (as well as that given by M. Demarçay above), viz., that ordinary active lead is there, induced by radium. Though according to experiences hitherto obtained the induced activity of bodies is relatively soon lost, it is yet possible that under the existing circumstances—minute quantities of lead, large quantities of radium, the long time of exposure to its influence, viz., one year—the resulting activity of the lead might be of longer duration as well as of greater intensity. This should be determined by research.

A decomposition into active and less active portions by chemical processes would not contradict these deductions, as I have convinced myself that ordinary lead with activity induced by radium becomes thus enriched. Debierne has already established this fact in the case of barium made active by actinium.

Hofmann and Strauss will see in the assumption of a new element and the above examination a confirmation of the identity of my substance with their "radio-lead," in order to enforce the conclusion of their latest publication, which runs:—"We are the first to have demonstrated the existence of a radio-active body closely allied to lead."

This statement necessitates the following explanation. I have shown that the Becquerel radiation of "radio-lead" is very slight, being considerably weaker than that of the pitchblende, which is the starting-point. The relatively strong action of the sulphate after only fifteen hours through the glass of the photographic plate is caused solely by the phosphorescence light of this substance; hence the negative result with the chloride, sulphide, &c., which either phosphoresce not at all, or insufficiently. If these light rays are kept back, after thirty hours no action takes place, and this only occurs after five days with glass 1·2 m.m. thick. Hofmann and Strauss consider the blackening of the photographic plate, not caused apparently by the action of light, as the result of the action of the Becquerel rays, and denote the substance thus tested as radio-active, as may be gathered from many examples in their publications. Hence none of these statements referring to the action through the glass of the plate are any criterion for radio-activity, because it is a question of radio-active substances. I first demonstrated that the phosphorescence light of the Becquerel rays is exhibited constantly; hence also the illusion of Hofmann and Strauss concerning the "latent-activity."

Let us add, for the better judgment of the strength of the activity of "radio-lead," that the first polonium preparations of Curie's were several hundred times as active as pitchblende. The Curies say in their paper that the new radio-active substances, including polonium and actinium, are at least 100,000 times as active as metallic uranium. (The ratio of metallic uranium to pitchblende is about 2:7). If, therefore, Hofmann and Strauss assert that the activity of "radio-lead" is only superseded by radium, they are quite mistaken. Even if it is allowed that the preparations left over for me to examine were only one-third of the strength of the newest, yet the intensity of the Becquerel radiation would be three times that of pitchblende at the most.

Accordingly, if "radio-lead" contains besides lead a strongly radio-active body, there can only be small traces of the latter. Under these circumstances it is incomprehensible how an atomic weight could be found widely differing from lead!

The determination of the "distinct spectral lines in the violet" is of the greatest importance for the existence of a new radio-active substance in "radio-lead." Spectrum analysis would also give the quantity of the new substance assumed by Hofmann and Strauss in their "radio-lead preparations," which they first looked upon as nearly pure, and tried to fit into the periodic system on the ground of their atomic weight determinations.

As my substance does not exhibit the same spectrum line, an identity with "radio-lead" is therefore excluded; and, in this respect, only an exact measurement of the "radio-lead" spectrum can procure certainty.

The simple fact that lead chloride and lead preparations from uranium ores are weakly active has long been known by Hofmann and Strauss, and also announced by them. On the other hand, the publications of the latter on "radio-lead," also on "radio-active lead," are so uncertain, and the results so confused in consequence of many alterations, that clearness on this side is desirable. Before this is done, the statement of Hofmann and Strauss quoted above, as well as their claim to priority, are futile.—*Berichte Deutschen Chemischen Gesellschaft*, January 18, 1902.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING JANUARY 31ST, 1902.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To CHARLES PERRIN, Esq., M.Inst.C.E.,
Water Examiner, Metropolis Water Act, 1871.

London, February 10th, 1902.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 216 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from Jan. 1st to Jan. 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 216 samples examined by us during the month, all were found to be clear, bright, and well filtered.

The rainfall at Oxford during the month shows a very serious deficit; the amount of rain measured was only 0·66 inch, the average for thirty-five years is 2·08 inches, leaving a deficit of 1·42 inches, or 68 per cent on the thirty-five years' average.

Our bacteriological examinations of 565 samples have given the results recorded in the following table; we have also examined 48 other samples, from special stand-pipes, wells, &c., making a total of 613 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 27 samples) ..	231
New River, filtered (mean of 140 samples) ..	15
Thames, unfiltered (mean of 27 samples) ..	4396
Thames-derived water, from the clear-water wells of eight Thames-derived supplies (mean of 309 samples)	30
Ditto ditto .. .. highest	258
Ditto ditto .. .. lowest	0
River Lea, unfiltered (mean of 27 samples) ..	233
River Lea, from the East London Company's clear-water wells (mean of 35 samples) ..	7

Of the 484 samples of impounded and filtered water supplied to London and examined bacteriologically during the past month, 20 samples, or 4·1 per cent, were sterile. Nine samples contained more than 150 microbes, while thirty samples, or 6·1 per cent, contained over 100 microbes per c.c. The mean number of microbes in the thirty excess samples was 143, against a corresponding mean of 185 in twenty-seven excess samples during December.

The impounding and filtration have been very satisfactory considering the time of year, and the extraordinary drought has prevented the increase in the organic matter which usually is characteristic of the Thames during the winter months.

We are, Sir,
Your obedient Servants,
William Crookes.
James Dewar.

ON PYRITE AND MARCASITE.*

By H. N. STOKES.

(Continued from p. 82).

VII. Data for Artificial Mixtures.

The following table gives the results for different mixtures, the numbers in the second column referring to the above-mentioned specimens of pyrite and marcasite respectively.

Values of p *for Mixtures of Pyrite and Marcasite.*

Per cent pyrite.	Specimens used.	*p*.	Mean value of *p*.
0	M 7	16·5	17·3 (18·0)
	M 10	17·4	
	M 12	18·0	
5	P 5; M 7	15·7	16·0
	P 5; M 10	16·5	
	P 5; M 12	15·9	
10	P 5; M 7	15·2	15·2
	P 5; M 10	15·2	
20	P 5; M 7	17·3	17·1
	P 5; M 10	16·9	
40	P 5; M 7	22·3	22·3
	P 5; M 10	22·2	
60	P 5; M 10	29·0	29·0
	P 4; M 10	29·1	
80	P 5; M 7	40·5	40·3
	P 5; M 10	40·2	
90	P 5; M 10	48·9	48·9
	P 4; M 10	48·9	
95	P 5; M 10	52·9	52·9
	P 5; M 10	53·0	
100	P 4..	60·3	60·5
	P 5..	60·8	

Discussion of the Curve.

The curve in Fig. 3 is based on the mean values of *p* thus obtained, with the exception of pure marcasite, where the more probable value 18 is used (see *ante*).

Influence of the Minimum; Indirect Determination of Small Amounts of Pyrite in Marcasite.—The most obvious features of this curve are the presence of a minimum point when the amount of pyrite reaches about 10 per cent and the increasing steepness as 100 per cent is approached. The minimum implies that a mixture of marcasite with a little pyrite has a less percentage of sulphur oxidised than has pure marcasite. This unexpected result, which is abundantly confirmed by numerous other preliminary data not published, may be partly due to the fact that pyrite uses up more ferric salt than does the equivalent amount of marcasite; the latter is therefore acted on by a solution weaker in Fe''' than if it alone were present, the effect of which is to lower the oxidation coefficient. Possibly there may also be an electro-chemical action between the two. A minimum also occurs in the curve for pyrite-galena, while pyrite-chalcopyrite shows a maximum. The existence of these points shows that it is impossible to predict, from a knowledge of the values of *p* for any two sulphides, whether the result of the contamination of one with a small quantity of the other will be to raise or to lower the oxidation coefficient. The variation of *p* from 16·3 to 18 for nearly pure marcasites is presumably due to small amounts of impurities other than pyrite. Practically these have but little effect on the determination of pyrite and marcasite, for, as seen in the table, the three samples of marcasite, though varying from 16·5 to 18, give essentially the same figures when mixed with even as little as 5 per cent of pyrite.

As a result of the minimum, any specimen in which *p* is about 18 or less may have one of two possible compositions. Which of these is actually present may be tested by adding one-ninth its weight of pyrite and determining the value of *p* for the new mixture. If depression is produced, the true percentage of pyrite lies to the left of the minimum; if elevation, to the right. In fact, the freedom of a marcasite from pyrite can be regarded as absolutely proved only when this precaution is taken. The determination of *p* in the original specimen is not necessary; the marcasite in question may at once be mixed with a known amount of pyrite and the original contents of pyrite thus determined. If x represents the percentage of pyrite in the original sample, then a mixture of 90 parts of this with 10 parts pyrite will give—

$$0{\cdot}9\,x + 10 = \text{per cent pyrite in mixture.}$$

The latter is found by one determination and the composition of the original sample deduced. An application of this method is given in Section X. (*prox.*) in the case of the marcasites from Garfield Tunnel (No. 20), Littmitz (No. 22), and Crow Branch Mine (No. 23). In the latter two cases the selected material showed no pyrite under a lens, although the specimen as a whole contained it in visible amount.

It further appears from the curve that small amounts o marcasite in pyrite can be determined with greater accuracy than small quantities of pyrite in marcasite. In the former case the determination can probably be made to 1 per cent with duplicate experiments and in the absence of notable amounts of other sulphides; in the latter it can hardly be more accurate than 2 or 3 per cent. It must be remembered, however, that there is a slight uncertainty close to both ends of the curve, due to the unknown influence of minute contaminations. In other cases, however, greater accuracy than is obtainable could hardly be desired.

VIII. Influence of Impurities.

When pyrite or marcasite is enclosed in rock, the extraneous material may be removed by digesting with hydrofluoric and hydrochloric acids; but small amounts of insoluble siliceous material can usually be neglected.

Miscellaneous Impurities.

The necessity of extreme care in preparing the material is emphasised above. Since $b > (c-a)$, an inspection of the equation—

$$p = \frac{8{\cdot}333\,b}{c-a} - 25$$

shows that the presence of iron salts or oxides, or access of air during the decomposition, affects the ratio—

$$\frac{b}{c-a}$$

in such a manner as to lower the value of *p*, this effect being greater with pyrite than with marcasite. Free sulphur exerts very little influence, and is, moreover, removed by the preliminary extraction with ether. Without influence are also sulphates, carbonates, or soluble silicates free from iron, insoluble silicates, quartz, and in general

* From the *Bulletin of the U.S. Geological Survey*, No. 186.

any substance neither contributing iron to nor effecting reduction of the ferric solution. Pyrrhotite and limonite are easily eliminated. Zinc blende, galena, magnetite, and hæmatite, if present in but small amounts and not enclosed in the fine particles of FeS_2, are more slowly removed by prolonged digestion with hydrochloric acid. All sulphides are more or less readily attacked, and those free from iron, since they do not contribute to $c-a$, should, it might be expected, raise the value of p. If present in traces, they do not appreciably affect the result, but if existing in large amounts, they introduce considerable errors, and abnormal results are to be explained by a qualitative analysis. In reality their influence cannot be always predicted, as pointed out above, and some experiments bearing on this point are given below.

Arsenopyrite.

The action of ferric solution on arsenopyrite, or on pyrites or marcasites containing arsenic in notable amounts, is very characteristic, in that the value of b is always greater than that of c. This is due to the fact that a portion of the permanganate represented by b is consumed in oxidising arsenious to arsenic acid. It may be further observed that in such cases a permanent end-reaction for b cannot be obtained, as the last portions of arsenious acid are oxidised but very slowly in the cold by permanganate. In this event the titration must be stopped at the first change of colour, and since arsenic acid is precipitated very slowly by hydrogen sulphide, it is necessary to conduct the gas through for several hours at a tempera-

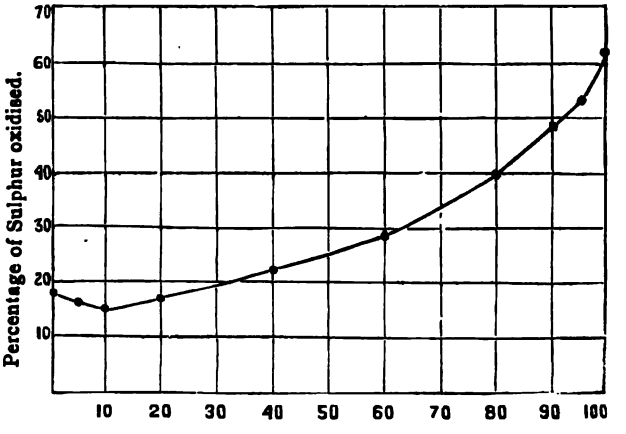

FIG. 3.—CURVE SHOWING OXIDATION COEFFICIENTS OF MIXTURES OF PYRITE AND MARCASITE.

The equation—

$$p = \frac{8{\cdot}333\ b}{c-a} - 25$$

applies, in its original sense of giving the per cent of sulphur oxidised, only to FeS_2. We may, however, use it in a broader way as simply indicating a certain relation between the permanganate values of the ferrous, ferric, and total iron, and in this sense it may be applied to any sulphide or other compound capable of reducing ferric salts, and is useful in instituting comparisons between these and FeS_2 either alone or in a mixture.

Hæmatite and Magnetite.

The presence of these is indicated during the extraction of the powder with 20 per cent hydrochloric acid if repeated portions of acid continue to be coloured yellow after heating. Difficultly soluble ferric silicates, of course, show the same result. As these iron oxides are scarcely soluble in the weakly acid ferric solution, their presence in small amounts is without appreciable effect.

Galena.

Galena is rather slowly attacked by the ferric solution, and the value of p is infinity. A mixture of galena and pyrite (p=60·8) containing 3 per cent lead gave—

$$p = 59{\cdot}5.$$

From this it appears that small amounts of galena exert a slight depressing influence, and that the curve for galena-pyrite varies from 60·4 to infinity, with a minimum near the pyrite end.

Nickel and Cobalt.

These show a very strongly elevating influence on the oxidation coefficient, as shown by specimens Nos. 27 and 32.

ture of about 80° in order to render the precipitation complete and to obtain a correct value for c.

An experiment made with the usual precautions, with carefully selected arsenopyrite gave—

a	39·50
b	56·90
c	45·84

On substituting these permanganate values we get—

$$p = 49{\cdot}8.$$

The actual sulphur oxidised, as calculated from these figures, assuming that all the arsenic is oxidised to As_2O_3, is 10·3 per cent, and in fact a notable sublimation of sulphur was observed. The data do not indicate whether any arsenic is liberated, or whether orpiment or realgar is formed, but no evidence of these could be obtained. More elaborate experiments would be required to decide whether under certain conditions they can be formed by the action of ferric salts on arsenopyrite.

A carefully prepared mixture of pyrite and arsenopyrite (2·17 per cent), containing 1 per cent arsenic, was oxidised with the following result:—

a	38·33
b	43·17
c	42·73

The value thus deduced for p, namely, 56·7, corresponds to that of a pyrite containing 2 per cent marcasite. As few pyrites contain as much as 1 per cent arsenic, it appears that in general the presence of arsenic will not interfere greatly with the marcasite determination.

Chalcocite and Bornite.

Chalcocite by itself gives an infinite value for p. The

influence of these minerals on pyrite is stated below. Both are very easily attacked by cold ferric solution.

Chalcopyrite.

As pointed out in the introduction, it is well known that chalcopyrite is more easily attacked by ferric solutions than is pyrite. An experiment with crystallised chalcopyrite, made with the usual precautions, gave—

a	37·44
b	44·01
c	44·11

whence—

$$p = 30{\cdot}0.$$

From these figures and the determination of the dissolved copper, it appears that not more than 2 per cent of the sulphur is oxidised, and that more copper than iron is dissolved (0·7 atom Fe to 1 atom Cu) from which it may be concluded that pyrite or marcasite is possibly an intermediate product of the oxidation of chalcopyrite by ferric solutions.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, February 6th, 1902.

Dr. ARMSTRONG, Vice-President, in the Chair.

MESSRS. E. L. Sherwood and A. G. Aston were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Frederic Guy Stirling Baker, Marryatts Lodge, Snaresbrook, Essex; Richard Blenkinsop, Garden Wharf, Battersea, S.W.; Alexander Brace, 10, Portland Terrace, Plumstead, S.E.; Fred Carrodus, Glen Fern Tower, Lansdowne Road, Wimbledon, S.W.; James Codrington Crocker, 2, Flynore Villas, Swansea; Gilbert Gunn, 209, Rochdale Road, Bury, Lancs.; Frank Eustace King, Woodbury, Polworth Road, Streatham, S.W.; A. Lionel Landau, 20, Highbury New Park, N.; Charles Henry Lockitt, 5, Harley Road, S. Hampstead, N.W.; Walter Ramshaw, 5, Fronwen Terrace, Brecon; William Scholes, 151, Ainsworth Road, Radcliffe, Lancs.; Arthur Lea Butler Tindall, 41, Sunnyside Road, Ilford; Edward John Wilkinson, 8, Blenheim Terrace, Leeds; Thomas A. Young, 318, Chetham Hill Road, Manchester; Cecil Revis, 77, King Street West, Hammersmith, W.

Of the following papers, those marked * were read:—

*10. "*Conversion of 1-Hydroxycamphene into β-Halogen Derivatives of Camphor.*" By M. O. FORSTER.

β-Bromocamphor, obtained by the action of bromine on 1-hydroxycamphene (*Proc.*, 1901, xvii., 245), yields camphor when reduced with zinc dust and acetic acid, and is hydrolysed by alcoholic potash, forming α-campholenic acid. The oxime remains indifferent to alcoholic potash, and is not converted into a nitrile under the influence of mineral acids; it yields a *benzoyl* derivative which crystallises from alcohol in silky needles melting at 71—73°.

β-Chlorocamphor, $C_{10}H_{15}OCl$, prepared from 1-hydroxycamphene and chlorine in acetic acid containing sodium acetate, crystallises from alcohol in long, slender prisms melting at 132·5: it has $[\alpha]_D = +39{\cdot}5°$ in chloroform, and $[\alpha]_D = +40{\cdot}7°$ in alcohol. The *oxime* is isomorphous with β bromocamphoroxime, and melts at 134°; the *benzoyl* derivative crystallises in silky needles melting at 86°.

β-Chloro-α-bromocamphor, $C_{10}H_{14}OClBr$, produced by the action of bromine on β-chlorocamphor, crystallises from petroleum in tabular aggregates of white prisms, and melts at 101°; it has $[\alpha]_D = +126{\cdot}5°$ in chloroform.

The *methyl ether* of 1-hydroxycamphene, $C_{10}H_{15}{\cdot}OCH_3$, formed when the hydroxy-compound is heated with methyl iodide and dry silver oxide, is a colourless, limpid oil which boils at 193—194°, has sp. gr. 0·9314 at 20°, and $[\alpha]_D = -27{\cdot}3°$. The *ethyl ether*, $C_{10}H_{15}{\cdot}OC_2H_5$, resembles the lower homologue, and boils at 203—204° bromine converts it into β-bromocamphor.

*11. "*The Influence of Temperature on Association in Benzene Solution and the Value of the Molecular Rise of Boiling-point for Benzene at Different Temperatures.*" By W. R. INNES, M.Sc., Ph.D.

The molecular rise of boiling-point at different temperatures was determined by introducing phenanthrene, benzophenone, and benzil into benzene, boiling under reduced, atmospheric, and increased pressure. A constant pressure was maintained during each series of experiments at reduced or increased pressure by means of an automatic, electrically controlled regulator. The molecular rise of boiling-point (r) was also calculated (a) from the heat of vaporisation by means of van't Hoff's formula $100r = RT^2/L$ (calculated A) and (b) from the rate of change of vapour pressure with temperature by means of the equation $100r = Mp\left/\frac{dp}{dt}\right.$ in which M is the molecular weight of benzene and p the mean pressure (calculated B). The calculated molecular rise and the mean experimental values found for concentrations up to 6/100 grm.-molecules per 100 grms. benzene are given in Table I.:—

TABLE I.

Temperature.	54°.	58°.	63°.	75°.	80°.	93°.
Calculated A ..	21·6	22·2	23·0	25·0	26·5	29·5
Calculated B ..	21·05	21·6	22·4	24·25	25·5	27·8
Phenanthrene ..	21·0	—	22·4	23·55	25·25	27·3
Benzophenone ..	20·17	—	21·6	23·0	23·8	26·2
Benzil	—	21·0	—	—	23·3	26·24

The molecular rise calculated from the heat of vaporisation is greater than that calculated from the vapour pressure, the difference increasing with increase of temperature. The values found, using phenanthrene as the dissolved substance, agree very closely with those calculated from the rate of change of vapour pressure. The closely related substances benzophenone and benzil give numbers for the molecular rise which are almost identical. The molecular rise for benzophenone is about 0·8 less than that for phenanthrene at the same temperatures.

Series of determinations were carried out at different temperatures with the abnormal substances benzoic acid, *o*-bromobenzoic acid, β-benzil monoxime, and dimethyl tartrate. It was found that the dissociation of the complex molecules increased with the temperature between 54° and 80° in every case. Between 80° and 93° there was an apparent decrease of dissociation at all concentrations with benzoic acid and β-benzil monoxime; *o*-bromobenzoic acid and dimethyl tartrate were more dissociated at 93° in dilute solutions, but in more concentrated solutions the molecular weights became equal to those at 80° with the former substance and greater with the latter.

The heats of dissociation (Q grm. calories) of β-benzil monoxime, *o*-bromobenzoic acid, and benzoic acid were calculated from the molecular weights at different temperatures and found to be of the same order as those of a number of typical dissociating gases. Table II. gives the values calculated; V is the volume in litres occupied by the benzene in which 1 grm.-molecule of the substance, calculated as double molecules, is dissolved:—

TABLE II.

Substance.	T_1.	T_2.	V.	Q.
β-Benzil monoxime	278	331	13	14600
" "	331	353	14	19900
o-Bromobenzoic acid . ..	331	353	8·18	7500
" "	331	353	12·3	5300
Benzoic acid	336	353	8·18	22800

The heat of dissociation of nitrogen tetroxide is 28900 cal., iodine vapour 28500 cal., acetic acid 20000 cal., and dimethyl ether hydrochloride 8600 cal.

Discussion.

In reply to questions by Dr. Travers, Dr. Innes said that several series of determinations of the molecular rise of boiling-point were made; there was very satisfactory agreement in every case. The series with abnormal substances at 93° was not repeated, but the fact that the molecular weight of all four substances was higher than might have been expected seemed to show that the anomalous results at this temperature are not due to ordinary experimental error, whilst from the values obtained for the molecular rise of boiling-point it appears that they are not due to a constant error in the method.

*12. "*The Magnetic Rotation of Ring Compounds; Camphor, Limonene, Carvene, Pinene, and some of their Derivatives.*" By W. H. Perkin, sen., Ph.D., F.R.S.

The remarkable differences between the magnetic rotations of saturated *closed* chain or ring compounds, such as the derivatives of tri-, tetra-, penta-, and hexa-methylene, and the open chain compounds of the aliphatic series, both unsaturated and saturated, were first referred to, the ring compounds having a *much lower* rotation than those which have unsaturated open chains, but the *same* composition; their rotation is also *smaller* than that of the saturated open chain compounds from which they differ in composition by $-H_2$.

On comparing the magnetic rotation of the mono- and di-carboxylic acids and ketones of the closed chain compounds with those of the corresponding saturated open chain aliphatic compounds containing the same number of carbon atoms, considerable discrepancies were observed in the amounts by which they differ from each other, which range from about 0·33 to 0·64, and this want of accord has hitherto been an enigma, but it is now found that this is due to the comparisons having been made between the wrong members of the two series. If tri-, tetra-, penta-, and hexa-methylene be taken as the first, second, third, and fourth members of the series, as they undoubtedly are, and are compared with the first, second, third, and fourth members of the aliphatic series, allowing for the difference of composition, $(CH_2)_3$, these discrepancies disappear. The differences between the rotations of these series are then found to be nearly uniform, and amount to about −0·60 in the mono- and di-carboxylic acids, ketones, and mono-chloro-derivatives of hexamethylene. In the ring hydrocarbons, this difference is, however, much larger, amounting to about 0·988.

The next point considered was the influence of the double or *bridged ring* formation, in which the ring formation from the open chain compound has taken place twice, and therefore with a loss of four atoms of hydrogen. Camphor is a compound of this class, and it was found in its case that the bridged ring affected the rotation to the extent of −1·290, or a little more than twice that of the single ring. In borneol, an analogous result was obtained, whilst menthol, which contains only a single ring, gave an amount analogous to the synthetical ring compounds.

Camphene also contains a bridged ring, which is unsaturated, and after allowing for this, it gives a difference of −1·337, which is not very different from that of camphor. The magnetic rotation of pinene, which is isomeric with camphene but differs from it somewhat in the character of the bridged ring, gave results very similar to those of the latter, but which are a little higher.

In the case of the limonenes, which also are isomeric with camphene but contain only a single unsaturated ring along with the unsaturated group $CH_3{\cdot}C{:}CH_2$ outside the ring, the rotations are very different, the value of the single ring formation, allowing for unsaturation, being practically the same as in hexamethylene, 0·988.

The rotations of chlorine and bromine derivatives of camphor and the relationships of nitro- and pseudonitrocamphor and camphoryloxime were considered, as well as the rotation of camphylamine, bornyl chloride, or pinene hydrochloride, and dipentene dihydrochloride.

The refraction values of borneol and camphor and its derivatives were also given.

*13. "*The Transport Number of very Dilute Solutions.*" By B. D. Steele, B.Sc., and R. B. Denison, B.Sc.

The object of this research was to see if it were possible to obtain by the investigation of sufficiently dilute solutions a value for the transport number for solutions of salts, such as calcium chloride, which would result in a constant specific ionic velocity when calculated from different salts of the same cation, thus bringing these salts into line with others, such as potassium chloride, to the ions of which definite specific velocities can be assigned. In order to test this question, it was first necessary to develop a method by means of which the transport number could be determined for very dilute solutions. The method employed is a simple modification of that originally used by Hittorf and lately modified by Noyes; it permits of the electrolysis of a practically unlimited volume of solution being carried out in a vessel of reasonable size.

Measurements have been made for calcium chloride, nitrate, and sulphate, and for potassium chloride, in concentrations of 1 grm. equivalent in 250 litres; and, by combination of the transport numbers found with the conductivity at infinite dilution, identical values are obtained for the velocity of the chlorine ion from solutions of the calcium and potassium salts, and for the calcium ion from solutions of the chloride and nitrate. The velocity of the calcium ion calculated from measurements of calcium sulphate solution is, however, about 3·5 per cent higher. The transport number of potassium chloride agrees with the best determinations made in solutions up to 1/10 normal, and confirms Kohlrausch's assumption that this remains constant with further dilution.

*14. "*An Investigation into the Composition of Brittle Platinum.*" By W. N. Hartley, D.Sc., F.R.S.

Having had to report upon the composition of brittle platinum in the form of fragments of pins used for dental purposes, their average weight being under 20 milligrms. each, a description was given of the method by which the absence of metallic impurities, and also of silicon, was proved spectrographically. The brittle and crystalline character of the metal was shown to be in all probability caused by minute quantities of phosphorus or carbon, and by re-melting in a lime crucible under the oxyhydrogen flame its malleability was greatly improved, thus confirming the accuracy of the conclusion arrived at.

The difficulty in deciding beyond question whether there were iron lines in the platinum, or platinum in the iron spectrum, or lines of some impurity common to both metals, was pointed out, as showing the importance for practical purposes of determination of wave-lengths made with the greatest accuracy attainable, particularly in the case of such spectra as these where large numbers of lines are so closely grouped together as to have approximately the same wave-lengths.

15. "*Tetrazoline.*" Part II. By S. Ruhemann and H. E. Stapleton.

The authors find that the action of methyl iodide on tetrazoline is complicated, and they described two of the substances which are formed. One of them crystallises in dark blue needles having the formula $C_5H_9N_4I_3$, and is readily decomposed by water with separation of iodine, whilst the other forms colourless, well-developed crystals, and is an iodide of the formula $C_5H_7N_4I$, which may be transformed into the corresponding chloride and into the platinichloride, $(C_5H_7N_4)_2H_2PtCl_6$. The authors gave reasons for their view that their iodide (or the chloride) is not the salt of methyl tetrazoline; their work, however, is not yet advanced enough to fix the constitution of these two compounds, or to explain the reaction by which they are formed.

(To be continued).

PHYSICAL SOCIETY.

Annual General Meeting, February 14th, 1902.

Mr. T. H. Blakesley, Vice-President, in the Chair.

The Officers and Council for the following year were elected as follows:—

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Prof. S. P. Langley and Prof. H. A. Lorentz were elected Honorary Fellows to fill the vacancies caused by the deaths of Prof. Rowland and Dr. Koenig. The President of the German Physical Society was elected an *ex-officio* Fellow of the Society.

The Secretary then read the President's Address. It commenced by giving some particulars of the life and work of Rowland, Koenig, Langley, and Lorentz. On January 11th a telegram was sent in the name of the Society to Prof. Hittorf congratulating him upon the jubilee of his professionate. The work of translation, revision, and production of an English version of Gilbert's "De Magnete" has been completed, and a copy of the book presented to the Society by the President. The remainder of the Address dealt with the refusal of the law of this country to recognise as valid matter for the granting of letters patent anything which may have been brought before any of the learned or scientific societies. In the United States a man may appeal to the fact of his having read such a paper in proof of his subsequent claim to receive a valid patent for his invention. The law of this country works very inequitably. As examples, the invention of the microphone by the late Prof. Hughes, the President's invention of the "Astigometer," and the invention of wireless telegraphy by Prof. Lodge were given.

An Ordinary Meeting of the Society was then held, at which Mr. Littlewood exhibited an Attwood's machine.

The Society then adjourned until February 28th.

CORRESPONDENCE.

THE VOLUMETRIC ESTIMATION OF MANGANESE.

To the Editor of the Chemical News.

Sir,—As Messrs. Ibbotson and Brearley "have never denied" the statement I made relating to the oxidation of ferrous salt by dissolved oxygen, it would appear that they must admit its truth. They have not, however, while still insisting on the use of ferrous salt, issued any caution on this head. There is no occasion to add hydrogen peroxide in large excess, and it is both unscientific and wasteful to add more reagents in any chemical operation than are necessary. Mr. Reddrop's extreme care in this respect has been more than justified in this series of articles.

My point relating to the "important rare metals" has been misinterpreted in their letter of January 31, 1902; my meaning is not very obscure.

The point in their last paragraph is doubtless explained by the fact that they have departed from the instructions, and have used 2·27 times as much spiegel as was recommended, and, further, the conditions of the experiment they describe are never realised in practice.—I am, &c.,

Hugh Ramage.

Cambridge, February 11, 1902.

NOTICES OF BOOKS.

Practical Electro-Chemistry. By Bertram Blount, F.I.C., F.C.S., Assoc.Inst.C.E., Consulting Chemist to the Crown Agents for the Colonies. Westminster: Archibald Constable and Co., Limited. New York: The Macmillan Company. 1901. Pp. 374.

The author's intention in compiling this book was to give an account of those electro-chemical processes which have already been, or are likely in the future to be, turned to industrial use. For the sake of conciseness little or no historical matter has been included, and, for the same reason, comparisons of electro-chemical processes with chemical or metallurgical methods giving the same results have been confined to showing their relative advantages, it being assumed that the reader has a knowledge of the older processes. The author has dealt with the relation between the output of any given process and the energy necessary for its proper working in a very complete manner, as well as with the practical advantages to be gained by the use of electro-chemical processes in certain cases.

The book is divided into eight sections or chapters. After the introduction and a description of the general principles of the processes, we come to the winning and electrolytic refining of such metals as copper, lead, gold, silver, nickel, cobalt, tin, antimony, and zinc; this is followed by the winning and refining of aluminium, magnesium, and sodium in igneous solution. We may here remind our readers that all electro-chemical operations are performed either by the analytical property of electric energy when passed through an electrolyte, either aqueous or fused, or by the heat produced when a current is passed through a bad conductor which is not an electrolyte.

The electric furnace devised by Moissan is next fully described, and its various uses explained, such as the production of chromium, molybdenum, tungsten, the carbides, silicides, and carbon boride.

In Section V. we have the electro-deposition of metals, such as coppering, silver, nickel, and gold-plating, and the electro-deposition of alloys. Processes for the production of alkalis, both by using a fused and an aqueous electrolyte, command a good deal of attention; amongst other products considered being caustic potash, the chlorates, hypochlorites, and perchlorates.

Section VII. is on the electrolytic manufacture of organic compounds and fine chemicals, the purification of sugar juice, electric tanning, &c.; while in Section VIII. and last, we find the various sources of power discussed; the cheapest form of power at present is that afforded by moving water, a large steam plant using cheap coal coming next. Water power is generally most abundant where it cannot well be utilised, while steam power is uneconomical. What is really wanted is a direct method of converting the energy stored up in coal into electrical energy; this problem, however, has yet to be solved.

We must congratulate the author on the success he has achieved in turning out a really sound work, which is at the same time well and clearly printed and indexed.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxiv., No. 4, January 27, 1902.

Certain Properties of the Radiation emitted by Radio-active Bodies.—Henri Becquerel.—The author concludes from a series of experiments that, in the radiation from uranium, either there does not exist the non-deviable and slightly penetrating portion of the radiation; or, if it does exist, it is not emitted with an intensity in proportion to the intensity of the deviable portion or of the same order of magnitude as in the case of radium radiation. A second series of experiments show that the deviable portion of the radiation of radium, identical with cathode rays, transforms white phosphorus into red. It is very probable that the very absorbable non-deviable portion might also be very active in effecting this transformation, but, on account of the necessity of preserving the radium, a glass tube has to be used to contain it, and this cuts off all the latter rays.

Vapour-tension of Hydrogen Selenide and the Dissociation of its Hydrate.—MM. de Forcrand and Fonzes-Diacon.—It is already known that the vapour-tension of hydrogen selenide is 760 m.m. at −42°. The authors now obtain other points on the curve of vapour-tension, and from these numbers are enabled to calculate the heat of volatilisation of hydrogen selenide. Six fairly concordant values were obtained whose mean is 4·74 cals. The existence of the hydrate of hydrogen selenide may be proved by filling a flask with the damp selenide and exposing it to a temperature of about +5° C. A colourless crystallised hydrate is seen, which has apparently the same stability as the hydrates of chlorine, sulphurous anhydride, and methyl chloride. The tensions of dissociation of the hydrate were measured.

Lithium Antimonide, and the Preparation of some Alloys of this Metal.—P. Lebeau.—Antimony and lithium easily combine, with evolution of a great deal of heat. The violence of the reaction prevents the possibility of practically obtaining a definite compound by direct union. On the contrary, by electrolysing a mixture of equal weights of lithium chloride and potassium chloride with a cathode of antimony, a well crystalline antimonide of lithium is produced. This substance is not easily fusible, and corresponds to the formula $SbLi_3$. This same method of operation applies to the preparation of a considerable number of lithium alloys.

Action of Copper Hydrate on Aqueous Solutions of Metallic Salts.—A. Mailhe.—The author showed in two preceding papers that tetracupric hydrate gives with aqueous solutions of chlorides, bromides, and metallic sulphates mixed basic salts. This particular action of tetracupric hydrate on solutions of metallic nitrates leads, in all cases when a mixed salt is produced, to a compound of the tetra-metallic type, isomorphous (except in the case of cadmium nitrate) with tetracupric nitrate, $Cu(NO_3)_2.3CuO.3H_2O$, to which type the others correspond. Péligot's blue hydrate reacts sometimes with solutions of metallic nitrates to give a crystalline compound, but more often a less interesting amorphous body is produced.

Glyceroarsenic Acid.—V. Auger.—Arsenic acid and glycerin react very strongly on each other, producing the ether acids, setting free one or two molecules of water, but the product obtained is immediately hydrolysed in contact with the free water. This excludes the possibility of a preparation of an arsenio-glycerate by the wet method.

MISCELLANEOUS.

Royal Institution.—On Tuesday next, February 25, Mr. W. H. Shaw, F.R.S., Secretary of the Meteorological Council, will begin a course of two lectures at the Royal Institution on "The Temperature of the Atmosphere, its Changes and their Causes"; and on Thursday, February 27, Sir Henry Craik, K.C.B., will deliver the first of two lectures on "Scotland's Contribution to the Empire." The Friday Evening Discourse on February 28 will be delivered by Professor H. A. Miers, his subject being "Gold Mining in Klondike"; and on March 7 Professor Becquerel, Membre de l'Institute, Paris, will deliver a Discourse on "Radio-Active Bodies" (in French).

MEETINGS FOR THE WEEK.

MONDAY, 24th.—Society of Arts, 8. (Cantor Lectures). "Personal Jewellery from Prehistoric Times," by Cyril Davenport.
TUESDAY, 25th.—Royal Institution, 3. "The Temperature of the Atmosphere—its Changes and their Causes," by W. N. Shaw, M.A., F.R.S.
WEDNESDAY, 26th.—Society of Arts, 8. "Recent Inventions in Weaving Machinery," by Prof. Roberts Beaumont, M.I.Mech.E.
THURSDAY, 27th.—Royal Institution, 3. "Scotland's Contribution to the Empire," by Sir Henry Craik, K.C.B.
—— Society of Arts, 4.30. "The Industrial Development of India," by Nilkanth B. Wagle, B.A.
FRIDAY, 28th.—Royal Institution, 9. "Gold Mining in Klondyke," by Prof. Henry A. Miers, F.R.S., &c.
SATURDAY, March 1st.—Royal Institution, 3. "Some Electrical Developments," by Lord Rayleigh, F.R.S., &c.

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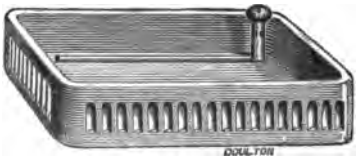

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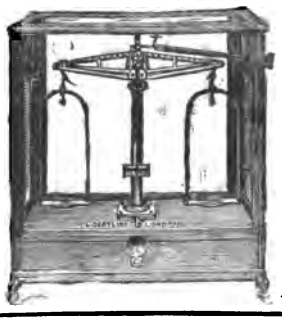

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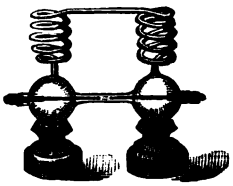

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THE CHEMICAL NEWS.

Vol. LXXXV., No. 2205.

THE STRATIFICATIONS OF HYDROGEN.*

By Sir WILLIAM CROOKES, F.R.S.

(Concluded from p. 87).

Stratifications in Pure Hydrogen.

The arrangement of the apparatus is shown in Fig. 5. The three hydrogen generators are called Nos. 1, 2, and 3. In No. 1, the gas is generated by the action of hydrochloric acid on zinc. This crude hydrogen is only used to drive out the air from the rest of the apparatus, and to remove the air dissolved in the liquids. When it had done its work, the generator was sealed off between Nos. 1 and 2, at A. It was considered that having the apparatus to begin with full of even somewhat impure hydrogen was better than starting with it full of air. The second and third generators contain at the bottom a pasty amalgam of mercury and zinc forming one pole, and a piece of platinum forming the other pole; the electrolyte is dilute hydrochloric acid. Platinum wires sealed through the sides of No. 3 carry the current from three Grove's cells to the interior. After the apparatus has had generator No. 1 removed, a large quantity of hydrogen is passed through from the second generator, with the object of replacing the impure hydrogen by some of a purer quality. When No. 2 is exhausted, it also is sealed off at B, leaving only the third generator with its drying tubes connected with the apparatus. Before sealing off No. 2, filling and exhausting is carried on until the hydrogen shows no impurity when spectroscopically examined in a capillary tube attached to the vacuum tube. The gas from the first and second generators bubbles first through strong caustic soda, C, C, C, to remove any acid carried over from the generators, then through strong sulphuric acid, D, to take away the bulk of the moisture, and thus save the drying tubes; it then passes through the purifying arrangements more especially connected with the third generator. Having sealed off Nos. 1 and 2, gas is evolved from No. 3 generator. Hence it passes through strong sulphuric acid in the tube H; then over a tube filled with granulated caustic soda, F; and next through a tube, G, tightly packed with phosphoric anhydride. H and I are two taps, having a reservoir, K, between them. When full of gas, H and I are closed, and the tubes L and M, after having been exhausted to a high point, can then be fed with limited amounts of pure dry hydrogen by slightly opening tap I, and closing it when equilibrium is restored between L, M, and K. N is a spiral of narrow glass tubing immersed in a beaker of ice and water. At O is a tap to keep mercury from diffusing into the pump if the apparatus has to be left all night. The vacuum tube, L, is provided with aluminium poles, and the tube M has the platinum poles made double for heating purposes, as shown in Fig. 3.

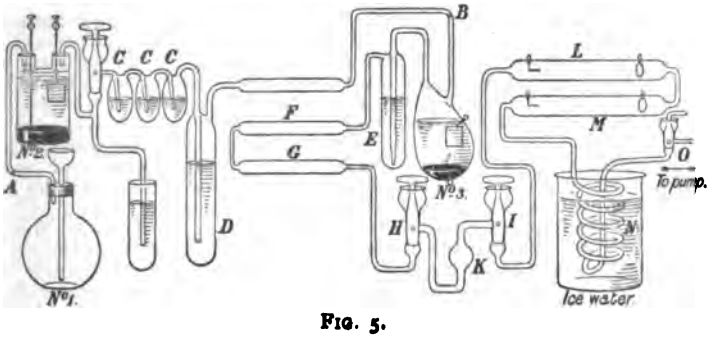


Fig. 5.

Hydrogen from the first generator was passed through the apparatus for two hours, when it was sealed off. The whole apparatus was exhausted to a high point, and No. 2 generator was set to work. Hydrogen was passed several times at full pressure through the apparatus for one or two hours, and then exhausted to the stratification point. During these operations the platinum terminals of one of the vacuum tubes were heated to full redness, and the current was kept on both tubes for some hours to drive off occluded gases.

Finally the second generator was sealed off, and hydrogen used from the remaining generator. After much washing out with hydrogen at the ordinary pressure, exhaustion and re-filling were continued, and finally the reservoir K was filled, both taps, H and I, being closed. The tubes were highly exhausted to the non-conducting point, and tap I opened and then closed, so as to introduce a little hydrogen. H was then opened and again closed, so as to equalise the pressure in I, and exhaustion proceeded to the stratification point. At first the strata were irregularly coloured with a suspicion of blue on one face, but as the operations just described were continued, the blue faces disappeared, the stratifications assumed a pure pink hue, and showed the hydrogen spectrum alone; no mercury was detected in any part of either tube.

From the first to the eighth filling the strata were pink with a trace of slaty blue colour on the faces next the negative pole. From the tenth filling the blue faces disappeared, and after the twentieth filling no trace of blue could be seen, and the spectrum of hydrogen alone was visible.

On examining the spectra of the stratified gas in the two tubes, each showed strongly the line spectrum of hydrogen; but while the spectrum in the platinum-poled tube showed pure red, blue, and green lines on a black ground, that in the aluminium-poled tube showed in addition the fainter hydrogen line spectrum in the yellow and orange. This result may be due to the greater surface exposed by the aluminium poles; it was not further examined.

Having at last succeeded in getting hydrogen free from mercury, experiments were instituted to verify the inference that the blue components of the blue and pink strata usually attributed to hydrogen were really due to the presence of a trace of mercury.

Origin of the Blue Component of Parti-coloured Stratifications.

I used an apparatus similar to the last, but with only one generator. If my idea was correct, that the mercury in the course of a few hours diffused into the hydrogen

* A Paper read before the Royal Society, February 6, 1902.

tube from the pump when it was not at work, there ought to be an access of blue faces to the pink buttons after the exhausted apparatus had been at rest. After filling with hydrogen and exhausting several times, a hydrogen vacuum was obtained showing no blue faces to the pink strata. The apparatus was then left all night, and the stratifications examined next morning. The blue colour to each face was now unmistakably visible. The refilling with hydrogen and exhausting was then continued. It was not possible in this way to get the tube entirely free from mercury, although it got less and less, as shown by the diminution of the blue faces.

Occasionally, when no mercury was present, a faint blue edging to some of the front pink strata was seen. This blue was too faint to show lines in its spectrum. After much searching the blue tint was traced to the phosphoric drying tubes. A clean tube was taken for stratifications, and sealed to the apparatus used in the last experiments. The whole was exhausted to a high point, and one of the phosphoric anhydride tubes was gently heated with a gas flame, the current kept going. Instantly a flood of blue light swept through the tube, and when concentrated in a narrow constriction the light showed a complicated spectrum which was not recognised; none of the characteristic lines of the phosphorus spectrum could be seen in it. The tube was cleared of the blue colour by introducing hydrogen and pumping it out a few times, and then hydrogen was introduced and exhaustion continued to the stratification point. The strata now were pink with no appearance of blue. Warming the phosphoric anhydride tube at once reproduced the faint blue edging to the pink discs. This blue colour was different both in tint and intensity to the blue colour produced by mercury, but it was too faint to show a spectrum except in the constricted part.

It is of importance to ascertain whether the body producing this blue colour can be removed from the phosphoric anhydride. The drying tube was again heated to the subliming point of the anhydride, hydrogen passed in, and the pump worked until the vacuum was almost non-conducting. The heating, passing in hydrogen, and pumping were several times repeated, the impurities diminishing each time. Ultimately a point was reached when, the tube being non-conducting, heating the phosphoric anhydride did not communicate any gas to the vacuum tube. At this stage the phosphoric anhydride still retained unimpaired its affinity for water. In any accurate experiment, therefore, the phosphoric anhydride tubes should have a preliminary heating in a vacuum to eliminate the impurity. This may be done with several tubes at a time, when they can be sealed at each end and preserved for future use.

It is thus seen that this blue glow is due to some impurity in the phosphoric anhydride. Likewise I have shown from the examination of its spectrum that it is not due to phosphorus. The glow probably is due to some intermediate oxide of phosphorus. In any accurate work with the mercury pump, where phosphoric anhydride is used as the drying agent, this source of impurity must not be overlooked.

An addition to the apparatus was made, a supplementary tube sealed on containing a grain of corrosive sublimate. This was used as being non-volatile at the ordinary temperature, but easily vaporised by heat. The experiment last described was continued, and immediately after the phosphoric blue edge appeared fresh hydrogen was let in and exhaustion continued till the faint blue was eliminated. The mercury salt was then heated, when immediately a rich blue edging appeared on the face of each pink stratification and the yellow lines of mercury shone out distinctly. Mercury blue is of a fuller colour than that of the phosphoric blue.

Altering the Colour of Stratifications.

In the *British Association Reports* for 1865 (Abstracts, p. 15), Mr. Gassiot describes the changes produced in the colours of the stratifications by introducing a water resistance in series with the vacuum tube. Having shown that the blue components of the stratifications are due to the presence of a trace of mercury with the hydrogen, experiments were commenced to ascertain what difference in the strength of the induced current would be necessary to alter the relative intensities of the pink and blue strata. Accordingly, I fitted up a resistance, shown in Fig. 6, consisting of a glass tube, A, A, 3 feet long and ⅝ inch diameter, nearly filled with distilled water. Through a cork, B, at the upper end of the tube a copper wire, C, C, passes, and by raising or lowering the wire either the whole resistance of the water or any fraction of it can be thrown into the circuit. The upper part of the wire is connected with one pole of the coil and the water is connected with the other pole by means of a small platinum wire sealed through the bottom of the tube.

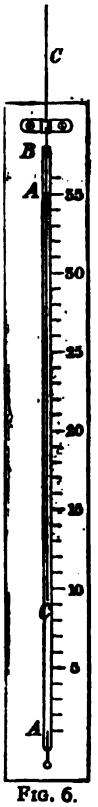


FIG. 6.

The wire was pushed down until it touched the platinum at the bottom, thus cutting out the water resistance. The strength of hammer-spring and the exhaustion were arranged to show good pink and blue discs. The wire was then gradually withdrawn, when the blue components gradually faded, and at a resistance of 6 inches of water the stratifications were all pink. Spectroscopic examination showed that in the parti-coloured state mercury was strongly present in all the blue components, together with the C hydrogen line; the mercury spectrum, however, being in excess. But when the water resistance was put in, and the buttons were all pink, mercury was still to be detected, but the hydrogen spectrum was more prominent. The green line of mercury was always the first to appear, and when the hydrogen became visible the first line to appear was the red.

That the change from parti-coloured stratifications to all pink is occasioned by an alteration in the intensity of the spark, is also shown by the fact that altering the make and break of the coil produces the same change as putting in the water resistance. Screwing up the hammer-spring so as to induce strong magnetism in the core and high potential of spark, gives pink and blue parti-coloured buttons. Weakening the spring, and thus weakening the magnetism, gives buttons which are all pink. I cannot, however, in this way cause the migration of the blue buttons to one single button in front. This phenomenon is produced only by an alteration of the exhaustion. Conversely, an alteration of the exhaustion will not change parti-coloured buttons to buttons of all one colour; this requires a change in electrical energy.

I consider the water resistance acts thus:—At the same rarefaction, hydrogen conducts the current better than mercury vapour. With a strong induction-spark at a certain degree of exhaustion, the conducting power of the rarefied hydrogen is not sufficient to carry the whole of the current; some of it, therefore, is conducted through the mercury vapour, when the intensity of the blue drowns the feebler colour of the hydrogen. When, however, a resistance of 6 inches of water (equal to about 500,000 ohms) is inserted, the current is so weakened that the hydrogen can carry the whole of it, and the blue of the mercury is not seen.

Conclusions; Chiefly Theoretical.

The phenomenon of blue faces on the pink discs is probably due to some such action as the following:—At the exhaustion necessary to give stratifications, there is a wide dark space round the negative pole. Here the

negative electrons (Radiant Matter), issuing from the pole with enormous velocity, have sufficient energy to clear a space in front of them to a distance varying with the degree of exhaustion.

Dr. A. Schuster considers that the discharge through mercury vapour in a vacuum tube, when quite free from air, will not give rise to stratifications, nor to the dark negative space (Dr. A. Schuster, "Experiments on the Discharge of Electricity through Gases," *Roy. Soc. Proc.*, vol. xxxvii., p. 318). My own experiments (*Journ. of the Inst. Electrical Engineers*, vol. xx., p. 44) show that the dark space will form in pure mercury vapour. Whichever view may be correct, there is no doubt that if stratifications in mercury vapour are not altogether unknown, they are much more difficult to produce than similar phenomena in hydrogen or other diatomic gases. At a certain critical stage of the exhaustion, when both hydrogen and mercury are present, I obtain both mercury and hydrogen strata.

The following experiments appear to show that the presence of hydrogen* has a considerable influence on the behaviour of mercury vapour in vacuum tubes. A tube with aluminium terminals had been attached to the pump for several days, and consequently mercury had diffused in and condensed on its sides. The tube was exhausted until, under the electric discharge, it phosphoresced of a brilliant green and was near the non-conducting point. It was heated very strongly with a Bunsen flame, when a faint pink luminosity was observed between the poles, the spectrum of mercury being absent. The tube was allowed to cool and a little hydrogen was introduced, when the usual pink stratifications appeared. On heating the tube to about the same temperature as before blue faces showed on the pink buttons, and the mercury spectrum was seen throughout the tube. especially in the neighbourhood of the negative pole.

It is known that in a vacuum tube, at an exhaustion aproaching the stratification point, any slight obstruction, such as constriction in the tube, or a series of wires sealed in, will cause luminous strata to hang round the obstruction. In a similar way, the hydrogen strata afford an anchorage, as it were, for the mercury, each hydrogen luminosity having a little blue glow of mercury hanging on to it; whereas, were there no hydrogen, no mercury stratifications would be seen.

The pink and blue luminosities show where the electrons and gaseous atoms meet; when the speed of the electrons is suddenly diminished, the shock throws the atom into greater vibration, which, being communicated to the ether, produces vibrations of definite wave-lengths, constituting the special spectrum of the atom. The dense mercury atom is not driven back so much as the lighter hydrogen atom—hence the blue front to the pink buttons. A very little difference in the exhaustion suffices to break the adhesion between the mercury and the hydrogen; then the mercury vapour diffusing along the tube meets the electrons from the negative pole and is swept back to the head of the hydrogen strata, and becomes apparent as a single button of blue light.

Radiant Matter. Electrons.

I have spoken of "Radiant Matter" and "Electrons" as if they were identical. Nearly twenty-five years ago I was led by experiments in highly rarefied tubes to assume the existence of matter in an *ultra-gaseous* state. Later, in a lecture delivered before the British Association at the Sheffield Meeting, 1879 (Chemical News, vol. xl., pp. 91, 104, 127), I first used the expression "Radiant Matter," or matter in the ultra-gaseous state, to explain the novel phenomena of phosphorescence, trajectory, shadows, mechanical action, magnetisation, and intense heat. "In studying this fourth state of matter," I said, "we seem at length to have within our grasp and obedient to our control, the little indivisible particles which with good warrant are supposed to constitute the physical basis of the universe. We have seen that in some of its properties radiant matter is as material as this table, whilst in other properties it almost assumes the character of radiant energy. We have actually touched the borderland where matter and force seem to merge into one another" (Chemical News, vol. xl., p. 130).

In twenty-five years one's theories may change, although the facts on which they are based remain immovable. What I then called "Radiant Matter" now passes as "Electrons," a term coined by Dr. Johnstone Stoney, to represent the separate units of electricity, which is as atomic as matter. What was puzzling and unexplained on the "Radiant Matter" theory is now precise and luminous on the "Electron" theory. Thus my early hypotheses fall into order by the substitution of one expression for the other. A chemical ion consists of a material nucleus or atom of matter constituting by far the larger portion of the mass, and a few electrons or atoms of electricity. The electrons are the same as the "satellites" of Lord Kelvin and the "corpuscles" or "particles" of J. J. Thomson.

Electrons probably leave the negative pole with a velocity nearly uniform, modified to a considerable extent by the degree of exhaustion, and to a less extent by the electromotive force behind them. Many experiments—the details I must leave to a future occasion—show that the liberated electrons do not behave as a gas, *i.e.*, they have not properties dependent on inter-collisions, mean free path, &c.; they act more like a fog or mist, are mobile, and carried about by a current of air to which they give temporary conducting powers, clinging to positively electrified bodies and thereby losing mobility, and settling on the walls of the containing vessel if left quiet.

On the other hand, the crowd of hydrogen or mercury atoms, by virtue of molecular motion and inter-collisions, act as gases. Whilst their *mean* free paths are conditioned by the degree of exhaustion, there may be amongst them a certain number of *actual* free paths differing widely on each side of the mean. Under the influence of the electromotive force, and at the right degree of exhaustion, these atoms arrange themselves in groups (see Note A), while the rushing swarm of electrons driven from the negative pole meet them and render them visible. According to J. J. Thomson the mass of an electron is about the 1/700th part of that of the hydrogen atom, and as these masses start from the negative pole in a vacuum tube with a velocity of the order of half that of light, it is easy to see that their heating, phosphorescent, and mechanical power must be stupendous.

The basis of the Electron, as I foreshadowed in 1879 in the case of Radiant Matter, is probably the same in all cases—the Protyle from which the chemical atoms were assumed to be formed (*Report of the Fifty-sixth Meeting of the British Association*, Birmingham, 1886, p. 568, Address to the Chemical Section).

On the two-fluid theory, the electrons constitute free negative electricity, and the rest of the chemical atom is charged positively, although a free positive electron is not known. It seems to me simpler to use the original one-fluid theory of Franklin, and to say that the electron is the atom or unit of electricity (see Note B). Then a so-called negatively charged chemical atom is one having a surplus of electrons, the number depending on the valency, whilst a positively charged atom is one having a deficiency of electrons. Differences of electrical charge may thus be likened to debits and credits in one's banking account, the electrons acting as current coin of the realm.

Notes.

A. In an Address delivered before the Institution of Electrical Engineers, January 15th, 1891, I gave an outline of a theory of stratifications in rarefied gases. The following quotation renders my meaning clear:—"If, in any much frequented street, at some time when the stream of

* Possibly another gas would do as well. Hydrogen was used because in this instance the hydrogen generator was attached to the tube.

traffic runs almost equally in both directions, we take our stand at a window from which we can overlook the passing crowd, we shall notice that the throng on the footway is not uniformly distributed, but is made up of knots—we might almost say blocks—interrupted by spaces which are comparatively open, we may easily conceive in what manner these knots or groups are formed: some few persons walking rather more slowly than the average rate slightly retard the movements of others, whether travelling in the same or in an opposite direction. Thus a temporary obstruction is created. The passengers behind catch up to the block and increase it, and those in front, passing on unchecked at their former rate, leave a comparatively vacant space. If a crowd is moving all in the same direction, the formation of these groups becomes more distinct. Hence mere differences in speed suffice to resolve a multitude of passengers into alternating gaps and knots. Instead of observing moving men and women, suppose we experiment on little particles of some substance, such as sand. If we mix the particles with water in a horizontal tube and set them in rhythmical agitation, we shall see very similar results, the powder sorting itself with regularity into alternate heaps and blank spaces. If we pass to yet more minute substances, we observe the behaviour of the molecules of a rarefied gas when submitted to an induction current. The molecules here are free, of course, from any caprice, and simply follow the law I seek to illustrate, and though originally in a state of rampant disorder, yet under the influence of the electric rhythm, they arrange themselves into well-defined groups or stratifications."—*Journ. of the Inst. Elect. Engineers*, vol. xx, p. 10.

B. "The theory of definite electrolytical or electrochemical action appears to me to touch immediately upon the *absolute quantity* of electricity or electric power belonging to different bodies. . . . And when comes the fact that the electricity which we appear to be capable of loosening from its habitation for a while, and conveying from place to place, *whilst it retains its chemical force*, can be measured out, and being so measured, is found to be *as definite in its action* as any of *those portions* which, remaining associated with the particles of matter, give them their *chemical relation*; we seem to have found the link which connects the proportion of that we have evolved to the proportion of that belonging to the particles in their natural state."—Faraday's "Experimental Researches in Electricity," par. 852.

"The equivalent weights of bodies are simply those quantities of them which contain equal quantities of electricity; . . . it being the ELECTRICITY which *determines* the equivalent number, *because* it determines the combining force. Or, if we adopt the atomic theory or phraseology, then the atoms of bodies which are equivalents to each other in their ordinary chemical action have equal quantities of electricity naturally associated with them."—*Ibid.*, par. 869.

"In former investigations of the action of electricity it was shown . . . that the quantity of electric power transferred onwards was in proportion to, and was definite for, a given quantity of matter moving as anion or cation onwards in the electrolytic line of action; and there was strong reason to believe that each of the particles of matter then dealt with, had associated with it a definite amount of electrical force, constituting its force of chemical affinity."—*Ibid.*, par. 1707.

(In all the above quotations the italics and capitals are Faraday's).

"It is therefore extremely natural to suppose that . . . every molecule of the cation is charged with a certain fixed quantity of positive electricity, which is the same for the molecules of all cations, and that every molecule of the anion is charged with an equal quantity of negative electricity."—Clerk Maxwell's "Treatise on Electricity and Magnetism," first edition, vol. i., 1873, p. 308.

"This definite quantity of electricity we shall call the molecular charge. If it were known, it would be the most natural unit of electricity."—*Ibid.*, p. 311.

"Suppose . . . we call this constant molecular charge, for convenience in description, *one molecule of electricity*."—*Ibid.*, p. 312. (The italics are Maxwell's).

"Nature presents us with a single definite quantity of electricity. . . . For each chemical bond which is ruptured within an electrolyte a certain quantity of electricity traverses the electrolyte, which is the same in all cases."—G. Johnstone Stoney, "On the Physical Units of Nature," British Association Meeting, 1874, Section A; *Phil. Mag.*, May, 1881.

"The same definite quantity of either positive or negative electricity moves always with each univalent ion, or with every unit of affinity of a multivalent ion."—Helmholtz, Faraday Lecture, 1881.

"Every monad atom has associated with it a certain definite quantity of electricity; every dyad has twice this quantity associated with it; every triad three times as much, and so on."—O. Lodge, "On Electrolysis," *British Association Report*, 1885.

ON RADIO-ACTIVE THORIUM.*

By K. A. HOFMANN and F. ZERBAN.

ACCORDING to the experiments of G. C. Schmidt, preparations of thorium possess the property of radio-activity. Rutherford has devised further methods of investigating this subject, and Debierne has then isolated from pitchblende a substance which was very strongly radio-active, and which behaved chemically like thorium. Since now the mineral bröggerite, which is rich in thorium, is related to pitchblende, it was to be conjectured that from bröggerite active thorium could be obtained in greater quantity than was possible from pitchblende. K. A. Hofmann and E. Strauss established the fact that the thorium ore isolated from bröggerite by the usual methods produced a strong effect on photographic plates. The thorium from clèveite and samarskite was also radio-active.

We have pursued these investigations and have found that the activity of the preparations of thorium from the above named minerals may be considerably augmented by fractional precipitation with concentrated solution of potassium sulphate, with potassium chromate, hydrogen peroxide, and sodium thiosulphate, by which means the activity is concentrated into those portions which are precipitated most readily.

On the other hand, on treatment with ammonium carbonate the active substance passes over into the more soluble fractions. Thus we hoped to be able to isolate the radio-active substance by repeated fractionation. But to our great surprise we found that, after keeping our preparation (as oxide in closed preparation tubes) for five months, its activity had very greatly diminished. For example, in June last year the most active thorium oxide from bröggerite, clèveite, and samarskite strongly blackened the photographic plate through glass, while in the following November the same substances, examined in the same way, produced only weak, and, in some cases, no perceptible effects. At the present time, *i.e.*, in January, none of these preparations produces any effect in ten hours through glass on the photographic plate, and most of them produce no effect through thin aluminium leaf. The decrease of activity may be conclusively proved by means of the electroscope.

Thus a bröggerite preparation, which was strongly active in June, discharged the electroscope in November in 1 min. 0 sec., while the electroscope without any radio-active substance lost its charge in 4 mins. 40 secs. The corresponding numbers in January were 3 mins. 45 secs. and 3 mins. 50 secs. Another bröggerite preparation

* Communication from the Chemical Laboratory of the Royal Academy of Science at Münich.

discharged in November in 1 min. 50 secs. (electroscope without radio-active substance, 4 mins. 40 secs.); in January in 2 mins. 30 secs. (electroscope without substance, 2 mins. 55 secs.). A clèveite preparation in November in 1 min. 10 secs. (electroscope without radio-active substance, 4 mins. 40 secs.); in January in 2 mins. 5 secs. (electroscope without radio-active substance, 2 mins. 15 secs.). A preparation from samarskite in November in 1 min. 30 secs. (electroscope without radio-active substance, 4 mins. 40 secs.); in January in 1 min. 50 secs. (electroscope without radio-active substance, 2 mins. 15 secs.). Another preparation from the same mineral in November in 3 mins. 45 secs. (electroscope without radio-active substance 4 mins. 50 secs.); in January in 3 mins. 40 secs. (electroscope without radio-active substance, 3 mins. 40 secs.).

According to these results we must assume that the thoria from bröggerite, clèveite, and samarskite possesses no primary, but only an induced activity. The supposition that the uranium always present in the minerals mentioned has exercised the original inductive action on the thoria has been confirmed. For we found that thorium preparations from a Brazilian monazite sand free from uranium were absolutely inactive immediately after their separation, both as regards the photographic plate, through thin aluminium leaf, through glass, and through caoutchouc, and also as regards the electroscope. This monazite sand contains no trace of uranium, but the thorium ore prepared from it became very strongly active after we had left it for a long time in contact with feebly active uranium. In order to meet the objection that the uranium employed by us contained another radio-active substance which mixed with the thorium during our experiment, the middle fraction, obtained by fractional crystallisation of the bröggerite-uranium as double salt with ammonium oxalate, was purified from all substances which are active in the above sense, by means of sulphuretted hydrogen, then with sulphuric acid, and finally with oxalic acid. The uranium thus obtained, when ignited to U_3O_8, showed itself feebly active.

Five grms. of this uranic-uranous oxide were ground fine together with 1 grm. of thoria from monazite sand, the mixture heated for four hours to 400°, and after cooling in a small closed preparation tube, left for fourteen days. Then it was dissolved by means of nitric acid, the thorium precipitated with oxalic acid, and ignited after careful washing. A similar experiment was performed with thoria from bröggerite, which had once been strongly active, but had become inactive through long keeping. Both thorium preparations now showed themselves to be strongly active, and even after four weeks they discharged the electroscope in 1 min. 5 secs. and 1 min. 15 secs. (electroscope without radio-active substance, 4 min. 0 sec.).

The uranium obtained from the filtrates of both thorium oxalates, when ignited to U_3O_8, appeared more feebly active than before. This result agrees with that of Becquerel, who showed that, by repeated addition of barium chloride and repeated precipitation with sulphuric acid, almost inactive uranium preparations may be obtained from the active uranium salt, while the precipitates of barium sulphate are active.

Our supposition that the activity of the thorium preparation from the minerals examined by us, *i.e.*, bröggerite, clèveite, and samarskite, is dependent on the presence of uranium, has been confirmed by the examination of other thorium minerals as well as by the above mentioned experiments. Bröggerite and clèveite contain a great deal of uranium (about 78 per cent U_3O_8), and strongly active thorium. Considerably less uranium occurs in samarskite (about 4 per cent U_3O_8), and the thorium ore obtained from it is correspondingly less active. Thorite with 50 per cent thorium and 10 per cent uranium is feebly active, orangite with 70 per cent thorium and 1 per cent uranium still more feebly active, and Brazilian monazite sand (free from uranium) is an inactive thorium preparation.—*Berichte der Deutschen Chem. Gesell.*, February, 1902.

A BIBLIOGRAPHY OF STEEL WORKS ANALYSIS.

PART XII.

By HARRY BREARLEY.

(Continued from p. 89).

IRON.

JOURNALS to end of 1900. The classification is—

I. SEPARATIONS FROM OTHER METALS.
II. GRAVIMETRIC ESTIMATIONS.
III. VOLUMETRIC ESTIMATIONS.
a. As Ferrous Salts.
b. As Ferric Salts.
IV. COLORIMETRIC ESTIMATIONS.
V. MISCELLANEOUS.

I. SEPARATIONS FROM OTHER METALS.

Lime and Magnesia.—

1635. ROSE (*C. N.*, ii., 291).—Add AmHO; any $CaCO_3$ formed is decomposed on boiling if AmHO salts are present.

1636. CALVERT (*C. N.*, ii., 328).—CaO is not separated with AmHO. Precipitate neutralised iron solution as basic succinate.

1637. GIBBS (*C. N.*, xi., 102).—Fe and Al are separated as acetate from Ca, Mg, Zn, and Ur.

1638. GUYARD (*C. N.*, xlix., 259).—Keeping Fe, Al, &c., in solution as citrate, Ca may be precipitated as oxalate and Mg as phosphate.

Uranium (see also 1637).—

1639. PISANI (*C. N.*, iii., 257).—Precipitate Fe with Am_2CO_3 and separate the little Fe in the filtrate with Am_2S.

1640. BURKER (*C. N.*, xxxviii., 276).—Process 1639 inaccurate. Precipitate with AmHO, weigh, reduce in H, and dissolve out the Fe with HCl (see 961).

1641. WALKER (*J. C. S.*, lxxiv., ii., 549).—Pour acid mixture into $NaHO+H_2O_2$; U in the filtrate. RHEINECK (*C. N.*, xxiv., 233).—Proceeds as 1637.

1642. CLASSEN (*J. C. S.*, xlviii., 192).—Fe deposited from solution containing large excess of oxalate leaves U in solution.

Beryllium.—

1643. SCHLEIER (*J. S. C. I.*, 1892, 713) and ATKINSON and SMITH (*J. C. S.*, lxx., ii., 220).—Fe precipitated with nitroso-β-naphthol.

1644. HAVENS and WAY (*C. N.*, lxxx., 285).—Be, Cr, and Zr are separated as in 1410).

Arsenic.—

1645. JANNASCH and KAMMERER (*J. C. S.*, lxx., ii., 221).—Pour HCl solution into $NaHO+H_2O_2$; As in solution.

Cadmium.—

1646. STORTENBECKER (*C. N.*, lxxx., 16).—The Fe is converted to ferrocyanide and the Cd electrodeposited.

Zirconium.

1647. MATTHEWS (*C. N.*, lxxix., 97).—Method based on insolubility of oxychloride of Zr, &c., in ether. Ce, Ti, &c., are similarly separated. Résumé of methods for separating Fe and Zr.

Thallium.—

1648. CROOKES (*C. N.*, vii., 194).—Reduce to FeO with hypo. and precipitate Tl with KI. Indicates 0·0002 grm.

Cerium (see also 1647).—

1649. GIBBS (*C. N.*, x., 195).—Add saturated Na_2SO_4. Ce, La, Dy are precipitated; Fe, Al, Gl, Yt, and Ur are in solution.

II. Gravimetric Estimations.

1650. Fresenius (*C. N.*, iv., 150).—Am_2S precipitates Fe slowly, but completely; AmCl assists; AmHO not harmful; ferric salts imperfectly precipitated.

1651. Blum (*J. S. C. I.*, 1888, 456).—Mix with tartaric acid, precipitate with Am_2S, dissolve in HCl, precipitate with AmHO, and weigh as Fe_2O_3.

1652. Koninck (*C. N.*, lxii., 19) and Meinecke (*J. S. C. I.*, 1888, 237).—Precipitate neutralised solution with nitroso-β-naphthol and ignite to Fe_2O_3. Al is separated, but not Cu, Co, and P.

1653. Cross. (*C. N.*, xxxvii., 26).—Extent of error introduced by precipitating $Fe_2(HO)_6$ in the presence of salts of the alkalis and alkaline earths.

1654. Meunier (*C. N.*, xx., 264).—Powdered meteoric stones digested with $AuCl_3$. Amount of Au deposited is estimated.

1655. Krug (*C. N.*, lxv., 69).—Résumé of methods for estimation of Fe (and Al) in the presence of P_2O_5. The following refer mainly to the valuation of agricultural products:—Ogilvie (*C. N.*, xxv., 277); Dyer (*C. N.*, liii., 51); Jones (*C. N.*, liii., 87; and lxv., 234); Mellon (*C. N.*, liii., 85); Glaser (*C. N.*, lxi., 181); Grüber (*C. N.*, lxiii., 146); Pellet (*C. N.*, xxxv., 268); Vincent (*C. N.*, lxx., 297); Gladding (*C. N.*, lxxiv., 112 and 146); Blattner and Brasseur (*C. N.*, lxxvi., 150); Lindet (*C. N.*, lxxvi., 212); and many others.

The following refer to electrolytic depositions:—

1656. Smith (*J. S. C. I.*, 1888, 693), Al and Ti do not interfere; Brand (*J. S. C. I.*, 1889, 1011); Rudorff (*J. C. S.*, lxiv., ii., 93); Heidenreich (*J. S. C. I.*, 1896, 744); Nicholson and Avery (*C. N.*, lxxiv., 91); and Avery and Dales (*J. S. C. I.*, 1899, 300 and 610).

III. Volumetric Estimations.

a. As Ferrous Salts.—

1657. Blunt (*C. N.*, viii., 54).—Very dilute solutions of HCl decompose permanganate. Rate of decoloration observed.

1658. Zimmerman (*C. N.*, xlvi., 12).—Presence of MnO salts prevents perturbing action of HCl in $KMnO_4$ titrations. See also Krutwig (*C. N.*, xlviii., 102); Thomas (*C. N.*, xlviii., 119) uses lead chloride for the same purpose; Hood (*C. N.*, l., 278) uses $MgSO_4$ or Am_2SO_4; Luke (*C. N.*, lviii., 186), sulphates of Ca, Co, and Mg have no value in this respect (see 1676.

1659. Moyance (*C. N.*, xx., 45).—Reduction to FeO best effected by amalgamated Zn. Best means of making $KMnO_4$ estimation stated.

1660. Kopp (*C. N.*, xxix., 242).—Uses $SnCl_2$ to accelerate the reduction by Zn.

1661. Beebe (*C. N.*, liii., 269).—Suspends the amalgamated Zn in a Pt basket.

1662. Donath and Jeller (*C. N.*, liv., 73).—Mix ignited $Al_2O_3+Fe_2O_3$ with Zn powder, heat, dissolve iron in H_2SO_4, and titrate. Process separates Fe from Al and Cr. Juptner (*J. I. S. I.*, 1894, ii., 497).—Uses Mg instead of Zn for ores, slags, &c.

1663. Carnegie (*J. C. S.*, liii., 468).—Reduces by filtering through Zn dust. Jones (*C. N.*, lx., 93).—Filters through pulverised Zn and illustrates its many advantages. See also Shimer (*J. C. S.*, lxxviii., ii., 50).

1664. MacIvor (*C. N.*, xxix., 246).—Hæmatites readily dissolve as FeO on heating with H_2SO_4 and Zn, and so avoid HCl in the $KMnO_4$ titration.

1665. Schmidt (*C. N.*, xlviii., 245).—Zn used to form FeO interferes with ferrocyanide indicator. $SnCl_2$ recommended.

1666. Storch (*J. S. C. I.*, 1893, 866).—Reduce to FeO with Cu and titrate with $KMnO_4$. Ignited Fe_2O_3 is soluble in 40 per cent H_2SO_4.

1667. Hart (*C. N.*, xxxiv., 65).—Reduce ore to metal in H, dissolve in H_2SO_4, and titrate with $KMnO_4$. Or, dissolve in HCl, add excess $SnCl_2$, and go back with I. Moissan (*C. N.*, xl., 89).—After heating in H, Fe may contain FeO and Fe_3O_4.

1668. Reynolds (*C. N.*, x., 228).—H_2S is a better reagent for forming FeO than either Zn or SO_2.

1669. Esilmann (*C. N.*, xxviii., 208).—Reduces Fe_2O_3 with Am_2S.

1670. Linossier (*C. N.*, li., 179).—Reduce with H_2S, destroy last traces by adding $HgCl_2$, and titrate with $KMnO_4$.

1671. Wells and Mitchell (*C. N.*, lxxiii., 123).—Reduce with H_2S; precipitated S no effect on $KMnO_4$ titration. Cu removed by filtration.

1672. Austen and Hurff (*C. N.*, xlvi., 287).—Résumé of Fe_2O_3 to FeO reductions; recommends Na_2SO_3. Boil until steam no longer decolorises $KMnO_4$. Reinsch (*C. N.*, v., 126).—Cu wire by a brown coloration on its surface will detect one-millionth part SO_2. Brown (*J. C. S.*, liv., 267).—Tests for SO_2 by paper soaked in ferric chloride and ferricyanide.

1673. Atkinson (*C. N.*, xlix., 117).—Bisulphite added to neutral solution best means of forming FeO. Errors due to Zn and $SnCl_2$. Speed of ferricyanide indicator and rate of decomposition.

1674. Clarke (*C. N.*, xvii., 234).—Fuse ore, slag, or cinder with bisulphate and cryolite, dissolve in HCl, reduce, and titrate with $K_2Cr_2O_7$.

1675. Reinhardt (*J. I. S. I.*, 1885, 299; 1889, i., 400; and 1900, i., 375).—Add $SnCl_2$ to hot acid Fe_2Cl_6, destroy excess with $HgCl_2$, add $MnSO_4$, and then H_3PO_4, so that Fe_2Cl_6 colour may not mask $KMnO_4$ end-reaction. See also Bya (*C. N.*, lvi., 279) and Mitchell (*J. S. C. I.*, 1894, 1096).

1676. Cady and Rudiger (*J. I. S. I.*, 1897, ii., 508).—$SnCl_2$ reduction; accurate $KMnO_4$ titration in the presence of $HgSO_4$.

1677. Namias (*J. I. S. I.*, 1892, i., 492).—Add excess $K_2Cr_2O_7$ to FeO solution, and a few drops of I and starch, and titrate with $SnCl_2$.

1678. Mixer and Dubois (*J. I. S. I.*, 1895, ii., 601).—Dissolve ore in HCl + $SnCl_2$, destroy excess $SnCl_2$ with $HgCl_2$, and titrate with $KMnO_4$.

1679. Mahon (*J. I. S. I.*, 1894, i., 617).—Pt carried by fusion flux into solution is reduced by $SnCl_2$ and leads to error.

1680. Mittenzway (*C. N.*, ix., 253).—Measure O absorbed by alkaline FeO solution by weighing the water which runs into closed operating bottle.

1681. Ross (*J. C. S.*, lxxii., ii., 193).—Add $K_2Cr_2O_7$ to FeO solution, and determine the excess by Baumann's process (1040).

1682. Parker (*C. N.*, xxii., 313).—Cu interferes with ferrocyanide indicator. Reduction of Fe_2O_3 with Zn would remove all the Cu.

1683. Hogg (*C. N.*, lix., 207).—Cu in Fe alloys is not dissolved by dilute acids (see 1534) and reduces Fe_2O_3 as it is formed in the titration. Dissolve in HCl+$KClO_3$, and reduce with SO_2.

1684. Rideal and Rosenblum (*J. S. C. I.*, 1896, 158).—Nickel gives a brown colour with ferrocyanide, which may mask the end-reaction with dilute FeO solutions. Jervis (*C. N.*, lxxvii., 133).—Mn acts similarly.

1685. Blum (*J. I. S. I.*, 1900, ii., 582).—The almost unfailing presence of V in tap cinder interferes with volumetric determinations; gives elaborate gravimetric method.

1686. Atkinson (*C. N.*, xlix., 117).—Means of standardising $K_2Cr_2O_7$. The C in steel used for the purpose leads to low results (see 119).

1687. Wellborn (*C. N.*, xx., 57).—$FeSO_4$ is preserved from oxidation by placing camphor (in paper)

therewith. Gawalowski (*C. N.*, xlvii., 165) uses pyrogallic acid similarly.

1688. Vogel (*C. N.*, xxiii., 142).—An exposure to direct sunlight for thirty seconds may cause ferricyanide to give a blue colour with ferric salts.

1689. Stock (*C. N.*, xxxix., 47).—A simple apparatus for effecting reductions out of contact with air.

1690. Johnson (*C. N.*, xlv., 287).—FeO solutions are oxidised the more readily if external air is excluded; suggests that $FeSO_4$ has ozonising action on enclosed air.

1691. Pattinson (*C. N.*, xxi., 268).—Hot $FeSO_4$ solutions exposed to the air to cool are not appreciably, if at all, oxidised.

1692. Willbur and Whittlesey (*C. N.*, xxii., 2).—Mix powdered silicate with CaF+HCl, heat in coal-gas, and titrate FeO with $KMnO_4$. Allen (*C. N.*, xxii., 57).—Estimates FeO by digesting ore or sand in a closed tube with HCl.

1693. Earley (*C. N.*, xxx., 169) and Dölter (*C. N.*, xxxix., 135).—Decomposes silicate with HF, and titrates with $KMnO_4$ or $K_2Cr_2O_7$.

1694. Pratt (*J. C. S.*, lxvi., 482).—Dissolves silicate in $HF+H_2SO_4$ in CO_2 in a Rose's crucible.

1695. Chester and Cairns (*J. C. S.*, liv., 196).—Use $AmF+H_2SO_4$ in CO_2 atmosphere.

1696. Penfield and Foote (*J. C. S.*, lxxvi., ii., 305).—Determine FeO in tourmaline by fusing with borax in an inert atmosphere and titrating H_2SO_4 solution with $KMnO_4$.

1697. Hillebrand (*C. N.*, lxxxii., 211).—Comparison of sealed-tube and HF methods of estimating FeO; the former often too high. See also *C. N.*, lxxviii., 106.

1698. Neumann (*J. S. C. I.*, 1887, 680).—Metallic Fe in slag, &c., is estimated by treating with acid and collecting the H evolved in a modified Lunge nitrometer.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, February 6th, 1902.

Dr. Armstrong, Vice-President, in the Chair.

(Concluded from p. 94).

16. "*The Solubilities of the Calcium Salts of the Acids of the Acetic Acid Series.*" By J. S. Lumsden, D.Sc., Ph.D.

It is well known that many calcium salts diminish in solubility with increase of temperature, and in order to obtain further information on this subject the solubilities of the salts of the acids of the acetic acid series have been carefully determined.

Calcium formate is anhydrous when in contact with its saturated solution, and gives a simple ascending curve of solubility. The calcium salts of the other acids of the series contain water of crystalisation, and all diminish in solubility with rise of temperature until a minimum point is reached, after which the solubility increases.

Of the salts of the normal acids only calcium acetate changes from one crystalline state to another between 0° and 100°, whilst both calcium *iso*butyrate and calcium *iso*valerate have double curves. With the exception of calcium formate all the salts which have been examined, when in contact with their saturated solutions at 100°, consist of crystals which contain one molecule of water.

The salts of the normal acids increase in solubility from formate to acetate and propionate, then decrease quickly with the growth in the number of carbon atoms, the salts of the *iso*-acids being more soluble than those of the corresponding normal acids.

17. "*The Equilibrium between a Solid and its Saturated Solution at Various Temperatures.*" By J. S. Lumsden, D.Sc., Ph.D.

The solubilities of the calcium salts of the acids of the acetic acid series were found to be all represented by curves convex to the temperature axis, indicating at first decrease then increase of solubility.

A curve of this shape is in no way anomalous, for by considering the action of heat on each of the factors which condition the equilibrium in a saturated solution, it is seen to be typically a normal curve.

The factors which produce equilibrium in a saturated solution are:—The affinity between solid and solvent, the thermal energy of the solid, and the pressure of the dissolved particles. By resolving the solubility curve, which is an exact representation of the resultant of these forces at different temperatures, into component curves representing the forces, the influence of temperature on each can be studied.

When each force varies directly as the temperature, the solubility curve is a straight line; when the rate is different it is a curve convex to the temperature axis.

Some experimenters have given curves of solubility concave to the temperature axis, but examination of several curves of that kind has shown that they have been produced by the joining of points on two convex curves, so cutting out the transition point.

By estimating the heat of solution at two points on a convex curve where the solubility is the same, it was found that the sensible heat was much less at the higher point. It was also found that the heat of solution at any temperature diminishes with the concentration, and that the direction of the solubility curve indicates the heat of solution in a saturated solution only, being positive on a descending and negative on an ascending curve.

18. "*On the Union of Hydrogen and Chlorine.* Part IV. *The Draper Effect.*" By J. W. Mellor and W. R. Anderson.

The momentary expansion which occurs when a mixture of equal volumes of hydrogen and chlorine is exposed to a flash of light is called, after its discoverer, the Draper effect.

This phenomenon is only produced when luminous rays impinge upon a mixture of approximately equal volumes of hydrogen and chlorine, but not upon chlorine alone, either under reduced or under ordinary pressures, or at temperatures between 15° and 100°. It is not produced when chlorine is mixed with steam, air, nitrogen, carbon dioxide, carbon monoxide, or methane. When the effect reaches a certain magnitude the mixture explodes. Although a few sparks bring about no measurable combination of the mixed gases, the joint effects of a great number of sparks at intervals of half-an-hour or of one hour is measurable, thus showing that chemical combination takes place during the Draper effect. The amount of combination depends not only upon the number but also upon the intensity of the sparks. The Draper effect appears to be due to some disturbance in the gas which attends chemical combination.

19. "*Note on the Constitution of certain Organic Nitrates.*" By C. R. Marshall and J. H. Wigner.

The authors criticise a theory recently advanced by Vignon and Gerin regarding the constitution of mannitol hexanitrate and allied substances. According to the French chemists, these higher nitrates reduce Fehling's solution strongly, whereas the lower nitrated compounds such as glycerol trinitrate have no reducing action whatever. In order to explain this they state that the terminal $-CH_2{\cdot}OH$ group of the higher polyhydric alcohols is converted, during nitration, into a hydrated aldehyde group, $-CH(OH)_2$, which under the influence of the nitrous acid formed during the reaction, is further changed to a

—CH(OH)ONO group. This, by the action of the alkali of the Fehling's solution, is converted to an aldehyde group which acts as the reducing agent.

Against this view it is urged that the amount of nitrite formed during hydrolysis would vary considerably in the different members of the series, which is shown not to be the case. It is further proved that these compounds possess only a slight reducing action, which can be explained on other grounds. The authors see no reason for departing from the view that these substances are nitric acid esters of the various alcohols.

20. "*Resolution of Trimethylhydrindonium Hydroxide into its Optically Active Components.*" By F. S. Kipping.

In continuing his work on hydrindamine (*Trans.*, 1900, lxxvii., 861; 1901, lxxix., 370, 430, and 442) the author has studied some of the salts of trimethylhydrindonium hydroxide,—

$$C_6H_4<\begin{matrix}CH_2\text{——}CH_2\\ CH(NMe_3\cdot OH)\end{matrix}>,$$

in order to compare their behaviour with that of the salts of the parent base.

dl-Trimethylhydrindonium iodide (*Trans.*, 1900, lxxvii., 469) interacts with silver *d*-bromocamphorsulphonate, giving dl-*trimethylhydrindonium* d-*bromocamphorsulphonate*, which can be separated into different fractions by crystallisation from a mixture of chloroform and ethyl acetate.

The most sparingly soluble fractions yield d-*trimethylhydrindonium* d-*bromocamphorsulphonate*,—

$$C_{12}H_{18}NO\cdot SO_2\cdot C_{10}H_{14}BrO;$$

it crystallises in slender needles, melts at 199—200°, and in dilute aqueous solution $[\alpha]_D = +70°$.

d-*Trimethylhydrindonium iodide*, $C_{12}H_{18}NI$, prepared from the preceding salt, crystallises from water in lustrous needles, decomposes at about 190°, and is very different from the racemic iodide in most respects; its specific rotation in dilute aqueous solution is $[\alpha]_D = +21{\cdot}9°$.

These results show that hydrindamine and trimethylhydrindonium hydroxide behave very differently towards one and the same acid.

21. "*Resolution of Methylbenzylacetic Acid into its Optical Isomerides.*" By S. F. Kipping.

Methylbenzylacetic acid, $C_6H_5\cdot CH_2\cdot CHMe\cdot CO_2H$ (α-methylhydrocinnamic acid), contains an asymmetric carbon group, is easily obtained in large quantities, and has recently been used for the preparation of methylhydrindone (*Proc.*, 1901, xvii., 181); in order to obtain an optically active acid chloride for various purposes indicated in the following note, the resolution of the acid was attempted.

Fractional crystallisation of the quinine salt gave the desired result; the salt of the *d*-acid is the more sparingly soluble in most solvents, and, after repeated crystallisation from dilute alcohol and ethyl acetate, is obtained apparently free from its isomeride.

Quinine-d-*methylbenzylacetate* crystallises in prisms, melts at 118—119°, and is very readily soluble in warm alcohol, but only sparingly so in boiling water; it has a specific rotation $[\alpha]_D = -113°$ in ethyl alcoholic, and $[\alpha]_D = -76°$ in ethyl acetate, solution.

d-Methylbenzylacetic acid is an oil at ordinary temperatures; the highest specific rotation which has been observed with different samples of the acid (without solvent) is $[\alpha]_D = +20{\cdot}3$. The sodium salt, purified by fractionally precipitating its alcoholic solution with ether, crystallises in colourless plates, and has a specific rotation $[\alpha]_D = +26°$ in dilute aqueous solution.

The chloride, prepared by treating the *d*-acid with phosphorus pentachloride and distilling under reduced pressure, is optically active, but probably contains considerable quantities of the *l* isomeride owing to partial racemisation having occurred.

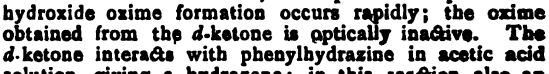
22. "d-*Methylhydrindone. The Formation of Oximes, Hydrazones, and Semicarbazones.*" By F. S. Kipping.

When the optically active methylbenzylacetyl chloride described in the preceding note is treated with aluminium chloride under suitable conditions (*Proc.*, 1901, xvii., 181) it yields an optically active methylhydrindone,—

$$C_6H_4<\begin{matrix}CH_2\\ CO\end{matrix}>CHMe;$$

the specific rotation of the crude product, which is probably a mixture of the *d*- and *l*-isomerides, is about $[\alpha]_D = +25°$ in methyl alcohol.

The optical properties of the ketone seem to undergo no change at the ordinary temperature, so that it consists entirely of the keto-form; in accordance with this conclusion, it does not give a colouration with ferric chloride. When heated at its boiling-point under the ordinary pressure for some time the ketone racemises, doubtless owing to keto-enolic isomeric change. It also racemises when treated with small quantities of sodium methoxide, sodium hydroxide, or sodium carbonate in alcoholic solution, and the gradual change in rotation can be followed with the polarimeter. There can be little doubt but that this racemisation is brought about by a keto-enolic transformation.



and these observations afford direct experimental evidence in favour of the accepted views regarding such changes.

Methylhydrindone and hydroxylamine do not seem to interact in absence of alkali, but in presence of potassium hydroxide oxime formation occurs rapidly; the oxime obtained from the *d*-ketone is optically inactive. The *d*-ketone interacts with phenylhydrazine in acetic acid solution, giving a hydrazone; in this reaction also an optically inactive product is obtained. These facts seem to show that oxime and hydrazone formation are preceded by the conversion of the keto- into the enolic form.

When the active ketone is treated with semicarbazide hydrochloride and sodium acetate in aqueous alcoholic solution it yields an optically inactive and an active semicarbazone; the former is probably produced owing to the presence of the *l*-ketone in the original substance, and the formation of an active product seems to show that in semicarbazone formation there is no previous transformation of the keto- into the enolic modification. These and other reactions of ketones are being further investigated.

23. "*Optically Active Methylbenzylacetic Acid.*" By A. Lapworth and W. H. Lenton.

In order to obtain further evidence on the question as to whether α-substitution in acids and ketones is the result of direct replacement of the hydrogen atom or is preceded by the formation of an ethylenic compound with the group ·Ċ:Ċ·O·, attempts were made to prepare an optically active acid which would undergo racemisation if the latter view were the correct one. For this purpose, methylbenzylacetic acid was thought to be the most convenient substance, as it is easily synthesised, and in its usual form is a solid at the ordinary temperature; the quinine salt of the acid was therefore fractionally crystallised from ethyl acetate until the melting-point and specific rotation of the least soluble portions remained constant.

Quinine d-*methylbenzylacetate* crystallised from ethyl acetate in well-formed needles which melted at 117°.

d-*Methylbenzylacetic acid*, $CH_2Ph\cdot CHMe\cdot CO_2H$, is a colourless oil; its specific rotation in 1 per cent ethyl acetate solution was $[\alpha]_D = +14{\cdot}9°$. The *ethyl* ester, under similar conditions, has $[\alpha]_D = +13{\cdot}2°$.

The active acid does not racemise appreciably at temperatures below 130°, but when its chloride is brominated at 80—90°, the product is optically inactive.

As the authors learn from Dr. Kipping that he has been

engaged on the study of active derivatives of benzylmethylacetic acid, they propose to leave its further investigation in his hands.

NOTICES OF BOOKS.

Practical Radiography; a Handbook for Physicians, Surgeons, and other Users of X-Rays. By A. W. ISENTHAL, F.R.P.S., and H. SNOWDEN WARD, F.R.P.S., Members of the Council of the Röntgen Society. Third Edition. London: Dawbarn and Ward, Ltd. 1901.

THE increasing application of Röntgen rays to medicine and surgery, and the many recent developments in the apparatus for their production, have rendered a third edition of this handbook necessary. To those engaged in radiography, or to any desiring sound information upon the subject, we can unhesitatingly recommend this third edition on account of its practical character, the authors having been actively connected with the application of Röntgen rays to surgery and other purposes from the time of their discovery. Between Chapters II. and XV. will be found a mass of valuable information. Induction coils, influence machines, interruptors, and other accessories to the production of the rays, are fully discussed, and descriptions are given of several little known pieces of apparatus, such as the Tesla oscillator, MM. Rochefort and Wydt's coil, and the closed magnetic circuit coil recently exhibited in London. The valuable mercury jet interruptor, introduced by Dr. Max Levy, is exhaustively described and illustrated, as is also Wehnelt's electrolytic interruptor. The history and development of the Crookes tube is given in detail with a description of the method of manufacture. The most valuable part of the book is undoubtedly that devoted to the *technique* of radiography, and here will be found full details of the methods of charging accumulators, exciting coils, screen work, photography localisation, and stereoscopic radioscopy and radiography.

We are glad to see that the application of X-rays to therapeutical purposes is only briefly touched upon; this field, although it promises great results, is in a very early state of development.

It is a pity to have to find fault with an otherwise trustworthy work, but some rather wild statements have found their way into the introductory and theoretical chapters. These need not have been introduced at all, but having been introduced they should have been carefully revised. On page 8 we are told that the vibrations of ether produce heat, light, magnetism, and *gravitation*, and that all these manifestations of ether vibrations can "be ranged in a sequence which we call the spectrum," and further down on the same page, "a solution of bisulphide of carbon, which to luminous radiations is completely opaque, offers no obstruction to dark heat waves." Here the solution of iodine in bisulphide of carbon is meant. Many other slips occur in this part, and in Chapter XV. appears a discussion of Professor J. J. Thompson's "corpuscle" theory that indicates confusion in the minds of the authors. However, apart from these minor faults, the book is undoubtedly one of the best that has yet appeared on the technology of radiography, and will be found of great value both to the operator and the student. There is a good index and it is well illustrated.

The Methods of Glass Blowing and of Working Silica in the Oxy-gas Flame. For the Use of Chemical and Physical Students. By W. A. SHENSTONE, F.R.S. Fourth Edition, Re-issue, with new Chapter. London: Longmans, Green, and Co. 1902.

THIS handbook is simply a reprint of that issued in 1886, with the addition of a short chapter on the method of working silica in the oxy-gas blowpipe for the formation of chemical and physical apparatus. Mr. Shenstone's success in working with quartz is so well known that any one desirous of fabricating quartz apparatus will gladly avail themselves of the practical experience detailed in this part of the book. The working of silica is not at all difficult, and with a little practice it will be found possible to produce rods, tubes, bulbs, and even small vacuum tubes from this material.

The Experimental Study of Gases; an Account of the Experimental Methods involved in the Determination of the Properties of Gases and of the more Important Researches connected with the Subject. By MORRIS W. TRAVERS, D.Sc., Assistant Professor of Chemistry and Fellow of University College, London. London and New York: Macmillan and Co. 1901.

THE above work being the outcome of researches carried out at University College during the past few years needs little further recommendation. It will be found full of descriptions of the apparatus and methods devised by the professors and assistants at that institution; many details of gas manipulation are described for the first time, well illustrated with original drawings. The collecting together with the latest corrections and modification of the mass of data that has been published at various times connected with the recently discovered gases of the atmosphere, will prove of considerable value to future investigators. We are sorry to note that for some unexplained reason all the promised plates of "*prism spectra of certain gases*," said to have been specially photographed for the book, are omitted, as are also Figs. 130 and 131; this is unfortunate as the photographed spectra in question are possibly the most valuable item in the description of the new gases.

There is an introductory preface by Professor Ramsay, a table of contents, and a good index.

The Cyanide Process of Gold Extraction. A Text-book for the Use of Mining Students, Metallurgists, and Cyanide Operators. By JAMES PARK, Professor of Mining and Director Otago University School of Mines, F.G.S., M.I.M.M., &c. Second English Edition, Revised and Enlarged. With Frontispiece, Plates, and Illustrations. London: Charles Griffin and Co., Ltd. 1901. Pp. 166.

So short a time having elapsed since the publication of the first English edition of this book there is obviously little we can add to the remarks we made in these columns when reviewing the previous edition (CHEMICAL NEWS, vol. xxxii., p. 157, September 28, 1900).

There are, however, a few points which merit attention. During the past two years the most noteworthy advances in the cyanide process have been in the treatment of slimes and sulphotelluride ores. The success of the new processes having been well-assured by actual experiment, the author has taken the opportunity afforded by this new edition to give a detailed description of the most up-to-date plants in South Africa, the United States, New Zealand, and Australia.

In Western Australia the oxidised ores are being replaced in depth by sulphide ores in which the gold is associated with tellurium, and the problem of treating these sulphotelluride ores is one of vital importance to many mines, especially to those of comparatively low grade.

There are two systems of treatment in use; the "raw ore" and the "roasted ore." In the former, introduced by Dr. Diehl, the ore is dry-crushed, then mixed with water, and, after amalgamation, ground to slimes in a large mill; the pulp is led to settling vats and the surplus water drawn off; the thickened pulp is then agitated with a solution of cyanide and bromide of potassium for twenty-four hours, then dried in a filter-press in which the dissolved gold is washed out. It is claimed that 93 per cent

of the gold can be recovered. The main features of the "roasted ore" treatment are drying, dry-crushing, roasting in a Ropp, Edwards, or other furnace, amalgamation in pans, cyaniding sands, if any, in vats, and the slimes in agitators; then filter-press separation, and washing of slimes. This latter process does not appear to us to be so simple or so economical as that of Dr. Diehl, as it necessitates much more "handling" of the ore. Both these methods are fully described, and plans of the plant used are given.

At the close of our remarks when dealing with the first edition of this book, while strongly recommending it, we ventured the opinion that "there will no doubt be a very large extension of mining operations when the pacification of the Transvaal has been completed, and the demand for really skilled managers and metallurgists will be greater than it has ever been before." We still adhere to that opinion, and though we were, like many others, a "little too previous," the very fact that more mills are getting to work every week round Johannesburg, and that the gold output for January last was over £250,000 in value, shows that the "good time coming" cannot now be very far off.

Mixed Metals, or Metallic Alloys. By ARTHUR H. HIORNS, Head of Metallurgical Department, Birmingham Municipal Technical School. Second Edition. Completely Revised and Enlarged. London: Macmillan and Co., Ltd. New York: The Macmillan Company. 1901. Pp. 445.

SINCE the first edition of this book was published in 1890 great advances have been made in the study of alloys, notably by Sir W. Roberts-Austen, Mr. Stead, MM. Charpy, Le Chatelier, Moissan, and other well-known chemists. And in this, the second edition, numerous references have been made to the published works of these and other eminent authorities. It is pointed out that the scientific study of alloys has not yet been sufficiently matured to permit of placing the subject on a logical scientific basis, and judging by the experience of different branches of applied chemistry, such as dissolution, fractional distillation, dissociation, &c., it will take many years to enable the new theories to be properly sifted, tested, and co-ordinated, as well as to connect such a scientific basis effectively with practical industry.

The principal chemical elements are first dealt with, and their classification into suitable groups; then the refractory materials used in making crucibles and in furnace construction, as well as the properties and uses of various fluxes, are described.

The alloys themselves are arranged in the following order:—(1) Copper alloys; (2) nickel alloys; (3) tin alloys; (4) lead alloys; (5) mercury alloys, or amalgams; (6) alloys of precious metals, including gold, silver, and platinum; (7) iron alloys; and (8) miscellaneous alloys, Under one or other of these headings will be found. besides the metals already named, alloys containing bismuth, antimony, arsenic, aluminium, nickel, cobalt, manganese, tungsten, titanium, sodium, potassium, and magnesium.

The book is intended essentially as a guide to manufacturers, workers, and students, and by the help of its copious index the reader will be able to find any particular alloy or mixture he desires with a minimum of trouble.

An Introduction to Chemistry and Physics. Two Vols. By W. H. PERKIN, Jun., Ph.D., F.R.S., and BEVAN LEAN, D.Sc., B.A. (Lond.) London: Macmillan and Co. New York: The Macmillan Company. 1901.

THIS second edition of a useful little book contains a good deal of new matter, and is divided into two volumes, the first providing a first year's course of physics, and the second dealing chiefly with chemistry. The book represents an attempt to follow the lines of historical development in initiating the young learners in the rudiments of these sciences, and thus it is hoped to foster the spirit of research always latent in the young, and to develop the power of reasoning from observation—the true aim of all scientific education. No one will be found to deny that the old way of teaching chemistry was unscientific, and often led to a distaste for a subject which entailed loading the memory with so many apparently disconnected facts; the new method minimises the efforts of memory, constantly calling the reasoning powers into play, and this book is a very good exponent of this system. The first volume is perhaps the better, being thoroughly practical and reliable throughout, while some parts are really excellent; for example, the chapter on "Graphic Representation" is most lucid and useful. The second volume will perhaps prove most valuable in the hands of a good teacher, whose aim should always be to make the learner find out as much as possible for himself. Many experimental details, such as the best way of collecting gases free from air, may be elicited from the learners themselves by dint of judicious questioning. It is, however, exceedingly difficult to dispense with the use of a text-book altogether in class-teaching, and this book can be thoroughly recommended as an excellent first course in physics and chemistry. The chapter on fuel and food-stuffs is practical and useful. and the account of Black's researches on chalk is specially good. Frequent problems and exercises intersperse the text, all probably well within the limits of time and capability of the learners, and a few examination papers enhance the book's usefulness.

Beiträge zur Chemischen Physiologie und Pathologie. Edited by FRANZ HOFMEISTER. Band I., 10-12 Heft. Braunschweig: Friedrich Viewig und Sohn. 1902.

THE January number of the *Journal of Chemical Physiology and Pathology* brings to an end the first volume, and contains the index. One paper of this number gives the results of the investigation and ultimate analysis of the albumens and cell membranes of bacteria and fungi, isolated by Krakow's new method recently communicated to the St. Petersburg Medical Society, and shorter papers deal with the subjects of the formation of sugars from protein substances, and the decomposition of guanidine by the animal body. Other articles of mainly physiological interest point to the great activity of investigators of this branch of science, in which so much work is still to be done.

CORRESPONDENCE.

THE SOCIETY OF PUBLIC ANALYSTS.

To the Editor of the Chemical News.

SIR,—In view of the press reports of the proceedings at a recent Dinner of the Society which is still incorrectly called the "Society of Public Analysts," we venture to ask you to give publicity to the following statement.

The title "Public Analyst" is legally applicable only to certain scientific officers, appointed by specified public authorities under the provisions of the Sale of Food and Drugs Acts, whose qualifications have been approved and whose appointments have been sanctioned by the Local Government Board.

At the present time only about 28 per cent of the members of the Society referred to are Public Analysts, owing to the fact that, as stated by its President, "the Society has widened its borders and now includes other analytical chemists" and persons concerned "with all branches of the chemical industry"—an expression of obviously wide application. The President is also reported to have said that "the great object of the Society was to ensure that its members were possessed of proper qualifications"—a

statement which, if left uncorrected, may cause serious misapprehension.

The Society in question is not, in any sense, a qualifying body, and its rules contain no specific requirements as to the scientific qualifications of candidates for admission to it, beyond the mere fact that each candidate must be recommended by three of the members, who testify "from personal knowledge" to his "scientific fitness." Indeed, one of the previous Presidents of the Society, when under examination before the late Food Products Committee of the House of Commons, found himself compelled to admit that membership of the Society did not imply the possession of any special qualifications.

The only qualifying body connected with the profession of Analytical and Consulting Chemistry is the Institute of Chemistry of Great Britain and Ireland, which was incorporated by Royal Charter in 1885, and which, under that charter, grants diplomas of Associateship and Fellowship to candidates who, having gone through prescribed courses of study and training, have passed the examinations conducted by the Institute.

From the facts stated above it will be seen that the Society referred to does not and cannot properly represent the Public Analysts of the country, that it cannot justifiably be described as a "Society of Public Analysts," and that it is in no sense a qualifying body.—We are, &c.,

(Signed)

J. Carter Bell, Public Analyst for the County of Cheshire, and the Boroughs of Salford, Birkenhead, &c.

Charles E. Cassal, Public Analyst for the City of Westminster, the Royal Borough of Kensington, the Metropolitan Borough of Battersea, and the County of Lincoln (Kesteven and Holland Divisions).

J. Kear Colwell, Public Analyst for the Metropolitan Boroughs of Finsbury and Holborn, and the Borough of Bedford.

C. Howard Cribb, Public Analyst for the City of Westminster, and the Metropolitan Borough of Fulham.

W. F. Keating Stock, Public Analyst for the County of Durham, and the Borough of West Hartlepool.

February 22, 1902.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxiv., No. 5, February 3, 1902.

New Synthesis of Formic Acid.—Henri Moissan.—The author has already shown that potassium and sodium hydrides, when slightly heated, react on carbonic acid gas, liberating carbon. The reaction produced with potassium hydride, when this latter is in the form of small crystals, causes sufficient heat to be evolved to bring the hydride to the point of incandescence. When sodium hydride is used, and a jet of carbonic acid is allowed to play on this hydride kept at the ordinary temperature, it is seen to suddenly burst into flame. This spontaneous combustion of an alkaline hydride in presence of carbonic acid leads the author to prepare formates by a new synthetic method.

Comparison between the Properties of Seleniuretted Hydrogen and those of Sulphuretted Hydrogen.—MM. de Forcrand and Fonzes-Diacon.—The authors measure various constants of the two gases, hydrogen selenide and hydrogen sulphide, using very pure specimens prepared by themselves. The constants are given in the following table:—

	H_2S.	H_2Se.
Temperature of ebullition (T) at 760 m.m. pressure (absolute)	211·4°	231°
Critical temperature (T_c)	373·2°	410°
T/T_c	0·566	0·564
P_c (critical pressure)	92 at.	91 at.
T' (fusion temperature)	187°	209°
D (density of the liquid at boiling-point)	0·86	2·12
PM/D (liquid molecular volume)	39·53	38·11
L (heat of volatilisation)	4230 cals.	4740 cals.
L/T (Trouton's relation)	20·01	20·52
C (heat of formation of hydrate)	16,340 cals.	16,820 cals.
t (temperature for which the hydrate has a tension of 760 m.m.)	273·35	281
S (solubility at +4·00°)	4·04 vols.	3·77 vols.
" " +9·65°)	3·60 "	3·43 "
" " +13·2°)	3·35 "	3·31 "
" " +22·5°)	2·75 "	2·70 "

Action of Lithium-ammonium on Antimony and Properties of Lithium Antimonide.—P. Lebeau.—Lithium-ammonium reacts on antimony, giving a compound having the formula $SbLi_3$, the same product as that obtained by electrolysis. This body dissolves in liquid ammonia and unites with it to form the compound $SbLi_3,NH_3$. Lithium antimonide is considerably less fusible than its constituent elements. This peculiarity is much the same as was observed for aluminium antimonide by Henri Gautier. Finally, this compound possesses very energetic reducing properties.

Oxyisopropylhypophosphorous Acid.—C. Marie.—A study of oxyisopropylhypophosphorous acid and its derivatives shows that it is a monobasic acid, to which, by analogy with M. Ville's oxybenzylhypophosphorous acid, the following constitutional formula may be attributed:—

$$\begin{matrix} CH_3 \\ CH_3 \end{matrix}\!\!>\!C\!<\!\begin{matrix} OH \\ PO \end{matrix}\!\!<\!\begin{matrix} H \\ OH \end{matrix}\cdot$$

Hydrolysis of Pyromucic Urethane.—R. Marquis.—M. Freundler prepared pyromucic hydrazide and azide, and established the formation of red condensation products by the action of dilute hydrochloric acid on the product of the action of the alcohol on the azide. The author applies this method of hydrolysis to the methylic and ethylic urethanes.

Action of Nitric Acid on Trichlorated and Tribromated Veratrols.—H. Cousin.—The action of nitric acid on the trichlorated and tribromated veratrols gives rise to mononitrated derivatives of tri-substituted veratrols. In this case the reaction is totally different from that obtained with the tetrachlorated and tetrabromated veratrols, because in that case tetra-substituted orthoquinones are formed by saponification of the ether functions and oxidation of the corresponding phenols. The action of nitric acid on the tri-substituted veratrols can be compared with the reaction of nitric acid on dibromated veratrol, which gives, as the author previously noticed, a mononitrated dibromated veratrol. Such a reaction is totally different from that obtained with the trisubstituted gaiacols, which, when treated with nitric acid, give diphenyl derivatives—compounds which contain no nitrogen and belong to the class of the quinones.

Some Derivatives of Glucamine.—E. Roux.—In a previous paper the author described a derivative of glucose, called glucamine, which possesses the formula $NH_2CH_2(CHOH)_4CH_2OH$. He now prepares a number of new derivatives of this base:—Cuproglucamine ($C_6H_{11}O_5NCu_2$), the picrate, chloroplatinate, chlorohydrate of pentacetylglucamine, hexacetylglucamine, benzalglucamine, glucamine-urea, glucamine-phenylurea, and pentacarbamic glucamine-phenylurea.

Action of Aluminium Chloride on certain Anhydrides in Chloroformic Solution.—Marcel Desfontaines

—The author finds that anhydrous aluminium chloride acts in all cases like sulphuric acid on the anhydrides presenting a tertiary carboxyl, such as the *aa*-dimethylglutaric and *aa*-dimethylsuccinic anhydrides.

MISCELLANEOUS.

Society of Chemical Industry.—As the Annual Meeting of the Institute of Chemistry is to be held on Monday, March 3rd, the next Meeting of the Section has been postponed from that date to Monday, March 10th, The Meeting will then be held at the Chemical Society's Rooms, Burlington House, Piccadilly.

Acetylene Lighting at a London Board School.—Mr. John C. Christie, of Aldgate, Gas Contractor to the London School Board, is at present fitting up the new Board School at Fulham Palace Road with acetylene. A portion of the school has been handed over for use, and the installation has proved most successful. The generators and purifiers used are of the Thorn and Hoddle Acetylene Co., Ltd., of Westminster, manufacture.

Small-pox Insurance.—Through the enterprise of Charles Letts and Co. (3, Royal Exchange, E.C.), of Diary fame, a small-pox insurance in combination with an excellent Note Book has been placed on the market for the nominal sum of one shilling. The insurance is for £100 in the event of the disease terminating fatally, with weekly allowance during illness (limited to five weeks). As the shilling also includes an excellent Note Book, with back loop, pencil, and elastic band, or, if preferred, a Card Case with stamp pockets, &c., the value is apparent, and particularly so to medical men. Unlike Lloyds and other insurance companies, Letts's coupons do not place doctors on an entirely different footing to the general public, demanding from the profession a largely increased premium for the risk covered. Our readers, therefore, cannot do better than to send to their stationers or to Messrs. Letts for a copy.

The Preparation of Tantalum by means of the Electric Furnace, and its Properties when thus Prepared.—Henri Moissan.—Powdered niobite mixed with sugar-charcoal, when subjected to the temperature of the electric furnace, yields a substance rich in niobium and tantalum. The metallic mass, treated first with a mixture of nitric and hydrofluoric acid, then with potassium fluoride, gives a mixture of fluotantalate and fluoxyniobate. The author separates these by Marignac's method, and decomposes the fluotantalate by sulphuric acid in a platinum capsule. After repeated purification, the tantalic acid is mixed with sugar charcoal and placed in the electric furnace: $Ta_2O_5+5C=2Ta+5CO$. The tantalum is obtained in the form of a brilliant metallic mass. The various properties of the metal which are examined show it to possess very strong reducing properties. Its reactions resemble those of niobium very closely; the increased reducing action being the only difference noticed between the two metals.—*Comptes Rendus*, cxxxiv., No. 4.

Investigation of Aluminium-Iron and Aluminium-Manganese Alloys.—Léon Guillet.—The author undertakes a systematic study of the alloys of aluminium with iron and manganese by reducing the oxide by aluminium in presence of excess of the latter. His experiments show —(1) That the limit of inflammability takes place when a mixture corresponding to the formation of the compound FeAl is used. However, in a Perrot's furnace the limit of inflammability may be extended until the theoretical formation of the compound $FeAl_4$ is reached. (2) All these reactions take place without loss, and they are slow. (3) The metallic globules are always very small, except in those experiments giving theoretically FeAl and $FeAl_4$. (4) The products prepared contain 2 to 15 per cent of free aluminium. (5) All the melts containing the metals in proportions between those giving theoretically FeAl and $Fe_{10}Al$ fall rapidly into powder. (6) All the melts obtained from the experiments comprised between the proportions corresponding to $FeAl_2$ and $FeAl_4$ give magnificent prismatic crystals whose length attains 6 or 8 c.m. The following compounds were isolated and analysed:—Fe_2Al_3, $FeAl_3$, Mn_2Al_3, $MnAl_3$, $MnAl_4$.—*Comptes Rendus*, cxxxiv., No. 4.

A New Reaction of Phosgene—B. Kühn.—The author has found, in collaboration with Spindler and P. von Gartzen, that phosgene is capable of breaking the bond between one atom of carbon and the tertiary aminic nitrogen; the valences set free are saturated by >CO and Cl-Cl. This phenomenon appears to bear a strong resemblance to hydrolysis, and probably depends on the dissociation of phosgene into ions. The authors have examined the reaction with the acetylised derivatives of the ortho-, meta-, and para-amidobenzylpiperidines which are split up by the action of phosgene at the ordinary temperature in the following manner:—

$$2CH_3.CO.NH.C_6H_4.CH_2.NC_5H_{10}+COCl_2=$$
$$=2CH_3.CO.NH.C_6H_4.CH_2.Cl+CO(NC_5H_{10})_2.$$

This decomposition allows of the preparation of the chlorides of the acetylised ortho-, meta, and para-amidobenzyls which are very susceptible to the reaction. The acetylised derivatives of the amidophenylpiperidines do not react in this manner, neither does benzoylpiperidine, which is, however, so easy to hydrolyse; for this reaction to take place it seems necessary for the piperidinic nitrogen to be united to the methanic carbon deprived of oxygen.—*Berichte*, vol. xxxiii., p. 2900.

The Bromisation of the Mixed Hydrocarbides.—A. Erdinger and P. Goldberg.—The authors have established the following method for the bromisation of the mixed hydrocarbides:—100 c.c. of HNO_3 (D = 1·4) are added to a solution of 25 grms. of the hydrocarbide to be bromised, in 100 c.c. of benzene; they then introduce, very gradually during two or three hours, an excess of bromide of sulphur; when the whole of the bromide has been added they decant the benzene solution, and shake well with caustic potash; the benzene is then distilled off, and the bromised derivative separated by distilling the residue with steam and fractionating over potash. This method gives, with a return of 85 to 95 per cent of theory, monosubstituted products with the exception of durol, with which they have always obtained, up to the present, a dibromised derivative. In this manner the authors have prepared bromobenzene (in this case an excess of benzene must be used), bromotoluene, the bromoxylenes, bromomesitylene, bromopentamethylbenzene, and bromonaphthalene; they also describe the dibromodurol which forms the exception.—*Berichte*, vol. xxxiii., p. 2883.

MEETINGS FOR THE WEEK.

MONDAY, March 3rd.—Society of Arts, 8. (Cantor Lectures). "Photography applied to Illustration and Printing," by J. D. Geddes, F.R.P.S.
—— Royal Institution, 5. General Monthly Meeting.
TUESDAY, 4th.—Royal Institution, 3. "The Temperature of the Atmosphere—its Changes and their Causes," by W. N. Shaw, M.A., F.R.S.
—— Society of Arts, 8. "Structural Colour Decoration of the Interior of Public Buildings," by Gerald C. Horsley.
WEDNESDAY, 5th.—Society of Arts, 8. "Sound Signals," by E. Price Edwards (Trinity House).
THURSDAY, 6th.—Royal Institution, 3. "Scotland's Contribution to the Empire," by Sir Henry Craik, K.C.B.
—— Chemical, 8. "The Slow Oxidation of Methane at Low Temperatures," by W. A. Bone and R. V. Wheeler. "Isomeric Additive Compounds of Dibenzyl Ketone and Deoxybenzoin with Benzal-*p*-toluidine, *m*-Nitrobenzalaniline, and Benzal-*m*-nitraniline" (Part III.), by F. R. Francis. "Mesoxalic Semi-aldehyde," by H. J. H. Fenton and J. H. Ryffel. "*m*-Nitrobenzoylcamphor," by M. O. Forster and F. M. G. Micklethwait. "Picrimidothiocarbonic Esters," by J. C. Crocker.
FRIDAY, 7th.—Royal Institution, 9. "Radio-active Bodies" (in French), by Prof. H. Becquerel, D.C.L., Ph.D., Membre de l'Institut, Paris.
TURDAY, 8th.—Royal Institution, 3. "Some Electrical Developments," by Lord Rayleigh, F.R.S., &c.

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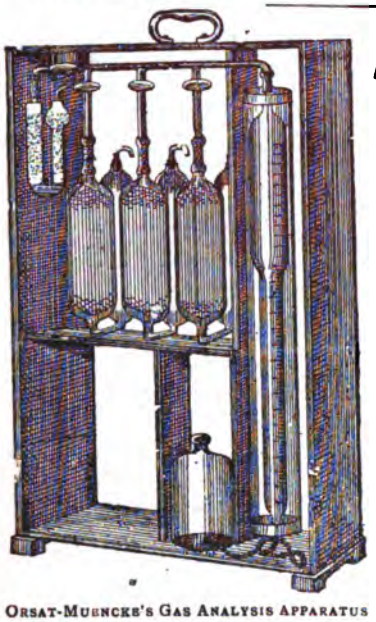

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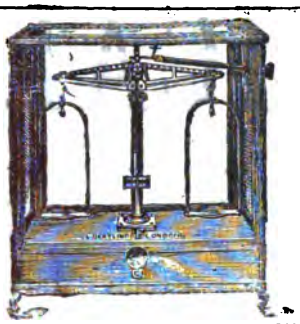

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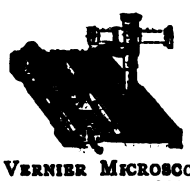

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THE CHEMICAL NEWS.

Vol. LXXXV., No. 2206.

RADIO-ACTIVITY AND THE ELECTRON THEORY.*

By Sir WILLIAM CROOKES, F.R.S.

Electrons emanating from radio-active bodies behave like material particles, and are impeded by the molecules of the surrounding medium, in contrast with ether waves, which are not thus affected except by absorption. It is not difficult to put these indications to test. A pair of shallow cells, A B (Fig. 1), 1·5 m.m. deep and 25 m.m. square, were made by cementing slips of glass to a thick glass plate. The cells were filled to the same depth with a radio-active substance chiefly containing actinium.† Over cell A was placed a piece of thick lead pipe, 28 m.m. high and 25 m.m. internal diameter, to ensure that any emanations from the active substance in A would be confined to the inside of the hollow cylinder. The radio-active substance in B was freely exposed to the air, save for a pillar of lead at C, to support the sensitive film. A sensitive film was laid horizontally over the cylinder and support C. On the film was a plate of glass, and cylinder and film were pressed together by heavy weights. The whole was covered in a light-tight box and put in a dark cupboard.

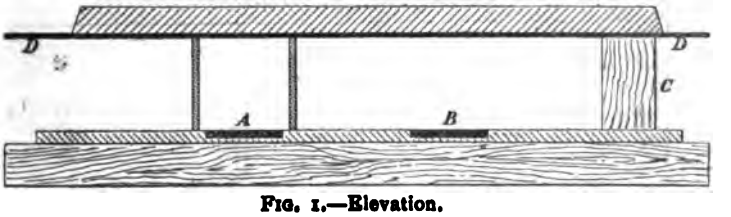


Fig. 1.—Elevation.

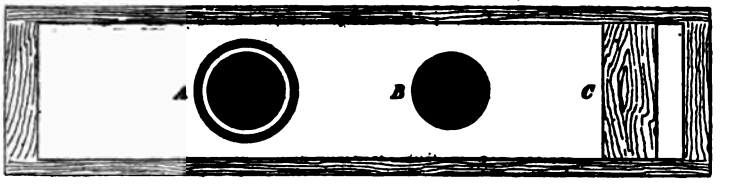


Fig. 2.—Plan.

At the end of forty-eight hours the film was removed and developed. There was a strong action shown over cell A (the one covered by the lead cylinder), but over B, the cell exposed to the air, there was no visible impression. Measured in Mr. Chapman Jones's "Opacity Meter" (*The Photographic Journal*, vol. xx., p. 86, Dec. 21, 1895), the results were:—

Image over cylinder—Opacity log.‡ = 0·79; Opacity§ = 6·17.

The experiment was repeated, using the same apparatus but a different preparation of actinium. In this case the exposure was for seventy-two hours. As before, there was a strong impression over cell A and none over cell B.] The figures were:—

Opacity log. = 0·89; Opacity = 7·71.

These experiments indicate that the electrons from the radio-active agent, chiefly actinium, partake of the properties of a fog or mist of material particles, capable of diffusing away in the free air like odoriferous particles, when not kept in by a thick metal screen.

A further experiment was now tried with the same apparatus, the agent a strongly active radium and barium bromide. This material being self-luminous, a sheet of black paper was placed immediately over it, so that nothing but emanations capable of passing through the opaque paper would be subject to experiment. After four hours' exposure in total darkness the film was developed. A good circular patch was obtained over cell A, and a faint diffused darkening showed over the rest of the film, darker at the spot immediately over cell B, fading away at the sides as the distance became greater. That this action was due to the material in the open cell B, and not to general fog over the plate, was seen by the clearness of the film where covered by the lead, and where shadows were thrown by the lead cylinder and pillar.

Circles of the same diameter were drawn round the dark impression over A, and round the darkest part of the impression over cell B. Measurements were taken of different parts of the spaces enclosed in these circles, and the mean of all these came out—

Circle over cell A—Opacity log. = 0·53; Opacity = 3·39.
Circle over cell B—Opacity log. = 0·32; Opacity = 2·09.
Ratio B/A = 0·62.

The experiment was repeated with the addition of a sheet of aluminium, 0·02 m.m. thick, under the black paper, the electrons now having to pass through both paper and metal before reaching the film. The exposure was for six hours, and the appearance on development was very similar to the last; a dark disc over the protected cell A, and a diffused action over the other part of the film, except in the shadow of the lead supports. Measurements as on the previous occasion gave the following results:—

Circle over cell A—Opacity log. = 0·78; Opacity = 6·03.
Circle over cell B—Opacity log. = 0·48; Opacity = 3·02.
Ratio B/A = 0·5.

A third experiment was tried with the same apparatus, using only the aluminium plate as a screen to cut off the luminous rays. The appearance on development was similar to the others, and the measurements of opacities were—

Circle over cell A—Opacity log. = 0·59; Opacity = 3·89.
Circle over cell B—Opacity log. = 0·29; Opacity = 1·95.
Ratio B/A = 0·5.

* A Paper read before the Royal Society, February 6, 1902.

† The body I called Uranium X in my Royal Society paper, May 10, 1900, has since proved to be M. Debierne's Actinium.

‡ The opacity logarithm represents the density of the image, absolute density being represented by 2·00.

§ The "opacity" is the whole number corresponding to the "opacity log." The "opacity" is directly proportional to the photographic energy acting on the sensitive surface.

Finally, I tried polonium subnitrate, which gives off emanations hardly capable of passing through any screen, and greatly obstructed by a few centimetres of air.

The apparatus was substantially the same as the one just described, with the modification that the lead cylinder was 12 m.m. high, and at the other end a rod of glass 12 m.m. high was used to support the film. The reduced height was chosen, experience showing that polonium emanations have great difficulty in penetrating many millimetres through air. The exposure was seven days, at the end of which time the film was developed. Over cell A a dark disc sharply defined the inside of the cylinder, while over cell B was a hazy diffused patch which to the eye looked much the fainter of the two. But measurements of patch A and of a disc over cell B of the same size as A, showed that the opacities in each case were practically identical, as shown by the following figures :—

Circle over cell A—Opacity log. = 0·74 ; Opacity = 5·49.
Circle over cell B—Opacity log. = 0·76 ; Opacity = 5·75.
Ratio B/A = 1·05.

A repetition of the experiment, taking the mean of five concordant results, gave the same opacities as before.

Without proving that the emanations from polonium are less material than those from actinium and radium, this experiment shows that their behaviour is entirely different as regards diffusibility through air. Whether this is due to the larger mass of the individual particles, or to the less distance they have to travel (12 m.m. as against 28 m.m. in the case of actinium and radium), or to some other cause, further experiments must decide.

M. and Mdme. Curie have shown that radio-activity is communicable from radium and actinium compounds to bodies such as lead, copper, glass, ebonite, and paraffin. After exposure to emanations from the radium compound, these bodies have the property of communicating temporary conductivity to the air and other gases, and of thereby discharging an electrified body.

When such charged bodies are exposed to the air, in a single day they lose the greater part of their activity. These phenomena are observed with different radio-active salts of radium, also with salts containing actinium ; but polonium compounds, even when very active, do not communicate the effect.

Dr. Rutherford shows that air which has remained for some time in the neighbourhood of thoria and then is carried in a current to a distance, retains its property of communicating radio-activity to other bodies. He explains these phenomena by supposing that thoria gives off a special kind of emanation capable of being conveyed by the air, and that this is the cause of the induced radio-activity.

To ascertain if the electrons or corpuscles from radium also possess the property of being carried along in a current of air, I fitted up an apparatus shown in Fig. 2. A, B, and C are three brass tubes closed at the lower end and cemented with paraffin to a wooden block. The upper ends were accurately ground to a level surface, and then coated with a thin layer of paraffin wax. Holes were drilled in B and C, to admit glass tubes, cemented air-tight into the cylinders, as shown in the figure. The upper end of the tube in B was closed with a plug of cotton-wool, and the outer end in C was connected to a water-pump, so that when the cylinders were closed at the top a current of air was drawn through B and C. As the radium compound was self-luminous, discs of thin aluminium foil were placed over cylinders A and B to cut off the luminous rays. A sensitive film was laid on the three cylinders over the aluminium, and it was tightly pressed down by a heavy weight ; the contact between the film and the tops of the cylinders being sufficient to make the whole air-tight. At the bottom of A and B a radium compound was placed, equal weights and equal surface in each. The whole was put into a light-tight box, and air drawn through. The cylinder A was used only as a standard. The air passing into B was expected to carry along with it some of the corpuscles emitted from the active material at the bottom ; and the inlet tube in C was turned up at the end, so that the stream of corpuscles-laden air should impinge on the surface of the centre of the film on C, and

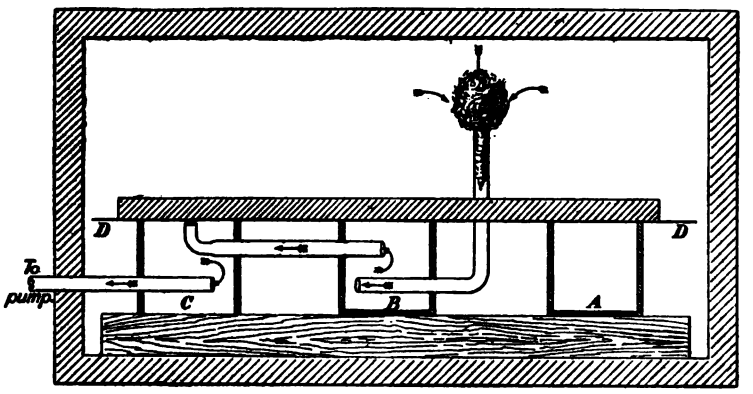


Fig. 2.—Elevation.

if it carried with it any radio-active properties the result should be seen on development, by the production of a dark patch. If, however, the air carried no corpuscles there would be no image on the sensitive film over C.

The experiment was continued for eleven hours, 120 litres of air having passed through in the time.

On development and measuring the resulting images the following figures were obtained :—

Circle over cell A—Opacity log. = 0·342 ; Opacity = 2·20.
Circle over cell B—Opacity log. = 0·178 ; Opacity = 1·51.
Circle over cell C—Opacity log. = 0·025 ; Opacity = 0·11.
Ratio B/A = 0·68.

The apparatus was slightly altered. Cells B and C were put nearer together, and a short wide brass tube replaced the turned-up glass tube formerly connecting them. The experiment was continued for forty-eight hours, during which time about 500 litres of air passed through the apparatus.

This repetition, allowing longer time, gave measurements as follows :—

Circle over cell A—Opacity log. = 0·964 ; Opacity = 9·20.
Circle over cell B—Opacity log. = 0·730 ; Opacity = 5·37.
Ratio B/A 0·58.

In this experiment a slight but measurable darkening took place over cell C, shown by an opacity log. of 0·025 and an opacity of 0·11. This shows that some few cor-

puscles have been able to pass through the wide tube from B to C, and act on the photographic film.

It thus appears that a current of air passed over the surface of a radium compound carries with it a certain proportion of the corpuscles. This is proved by the diminished photographic action in the second cell, slightly confirmed by the evidence that some few of the corpuscles so carried away get to the sensitive film on cell C. Judging from our slender knowledge of the properties of free electrons, it is highly probable that they will not easily turn a corner, but cling to the sides of the tube through which they are being led. On the other hand, the constant collisions with the atoms of air may reduce their initial mobility almost to a vanishing point before they have travelled along the tube between B and C, and then they would be carried along with the air.

The experiment was repeated, using a preparation of actinium (Uranium X). It was kept going for seventy-two hours, during which time 750 litres of air were drawn through the apparatus. On development and measurement the following results were obtained :—

Circle over cell A—Opacity log. = 0·99 ; Opacity = 9·78.
Circle over cell B—Opacity log. = 0·67 ; Opacity = 4·68.
Circle over cell C—Opacity log. = 0·25 ; Opacity = 1·78.

Thoria prepared by the ignition of Dr. Knöfler's highly purified thorium nitrate (*Roy. Soc. Proc.*, vol. lxvi., p. 421, May, 1900) was now tested in a similar apparatus to the one last described, the only alteration being the removal of the covering aluminium plates, thoria not being self-luminous. The experiment was continued for one hundred and eight hours, 1125 litres of air being drawn through the cells during the time.

Measurements of the developed images gave the following results :—

Circle over cell A—Opacity log. = 0·306 ; Opacity = 2·02.
Circle over cell B—Opacity log. = 0·260 ; Opacity = 1·82.
Circle over cell C—Opacity log. = 0·046 ; Opacity = 0·10.

Here also the results agree with those tried with radium and actinium compounds, that corpuscles are carried by a current of air from cell B, through the connecting tube to cell C. They also confirm those of Dr. Rutherford—who finds that thorium emanations travel in a current of air while retaining their activity—and of P. Curie and A. Debierne, who show that induced radio-activity can be transmitted through capillary tubes of an internal diameter of 0·1 m.m. and 75 c.m. in length, bent once at right-angles.

I have not obtained, however, a similar result with the emanations from hydrogen peroxide. As shown by Dr. Russell, this substance has a strong action on a sensitive photographic plate. The emanation from a bottle half full of hydrogen peroxide acts strongly on a sensitive film laid over the open mouth of the bottle for twenty-four hours, while there is no action in seventy-two hours if a U-shaped tube passed through the cork of the bottle and the sensitive film is put close to the open end of the tube. Dr. Russell tells me his observations confirm my experiments.

A highly active self-luminous radium compound loses some of its power on long exposure to the ordinary air of the laboratory. Ignition to red heat restores, however, its self-luminosity, and when sealed in a vacuum its activity remains as great as at first. When enclosed in glass the glass soon assumes a pink colour, which is not superficial, as already observed by M. and Mdme. Curie. By immersing a section of the glass in a liquid of the same refracting power as itself, the colour is seen to penetrate below the surface. If, however, the radium compound is sealed *in vacuo* in a *quartz* tube no coloration takes place, and I can detect no diminution of energy even in twelve months.

Electrons from radium will pass through aluminium and a considerable length of air and affect a sensitive film.*

* Using an active compound of radium, I have obtained an impression on a sensitive film through a penny-piece,

Experiments on this point were tried with polonium, and it was found that air offered great obstruction.

Three shallow cells 17 m.m. in diameter and 2 m.m. deep were filled with polonium subnitrate, the same quantity in each cell, the surfaces being levelled. Over these cells were fixed three pieces of lead pipe of lengths of 1, 2, and 4 inches, so that each cell of polonium had its emanations confined to its own tube. Sensitive films were put over each tube, and the whole kept in total darkness for 144 hours. At the end of that stretch of time the films were developed. No image was seen on the film 4 inches from the polonium, a faint image was perceptible on the film 2 inches off, and a stronger one on the film 1 inch off. Measured in the opacity meter the figures were :—

Over 1 inch tube—Opacity log. = 0·18 ; Opacity = 1·51.
Over 2 inch tube—Opacity log. = 0·04 ; Opacity = 0·11.

A repetition of this experiment, using tubes of glass instead of lead, gave almost identical results.

The electron theory explains a fact which has long puzzled experimentalists. It is well known that if a coin is laid on a sensitive plate in perfect darkness and connected with one pole of an induction coil for a few seconds and then developed, an image can be obtained of the raised parts of the coin. This has generally been explained by saying that the electrified stream of air, or the "brush discharge," affects the film like light.

But Mr. F. Sandford (*Nature*, vol. lv., p. 485) shows that coins embedded in the centre of a block of paraffin 2 c.m. thick, where they could not send off streams of electrified air, can still be photographed by means of the induction coil. Under these circumstances it is probable that electrons are the agents, as electrons will easily pass through paraffin wax from the coin to the sensitive plate, when the coin is connected with the negative pole of an induction coil, the other pole being connected with a metal plate placed below the wax block.

Hitherto we have been dealing with negative electrons—a free positive electron at present is unknown. In a paper communicated to the Royal Society, December, 1900 (*Phil. Trans.*, A, 1901, vol. 196, p. 525), the Hon. R. J. Strutt offers a suggestion as to positive ions which in a satisfactory manner appears to explain much that hitherto has been left doubtful, not to say contradictory.

He adopts the generally recognised theory that the deflectable Becquerel rays consist of a stream of negative corpuscles with enormous velocities proceeding from the radio-active body. But there are two kinds of Becquerel rays, one deflectable and penetrating, the other non-deflectable and easily absorbable. Mr. Strutt considers that these non-deflectable rays are positive ions moving in a stream from the radio-active body.

He says :—"We know that the positive ions in gases carry the same charge as the negative, and that they have an enormously greater mass. Unless, therefore, their velocity is smaller out of all proportion than the negative ions, it is to be expected that they will be much less easily deflected by the magnet. . . . Next it may be noticed that the smaller penetrating power would be well accounted for by the size of the positive ions, which would, of course, make more collisions with the molecules of the surrounding gas than the much smaller negative ions."

Of the three radio-active bodies, radium, actinium, and polonium, actinium appears to emit corpuscles almost entirely of the penetrating, deflectable kind, polonium rays of the non-deflectable, non-penetrating kind, whilst radium emits rays of both kinds.

On the above hypothesis corpuscles from polonium might consist of the heavy positive ions; to test the accuracy of this inference experiments are now in progress.

Some curious and far-reaching inferences may be drawn from Mr. Strutt's view, supposing it to be correct, that positive as well as negative corpuscles will fly off from a radio-active body. In a paper "On Electrical Evaporation" (*Roy. Soc. Proc.*, June, 1891, vol. l., p. 88) I showed that many bodies, such as silver, gold, platinum, &c.,

usually considered non-volatile at ordinary temperatures, easily volatilise in a vacuum if connected with the negative pole of an induction coil, remaining fixed when connected with the positive pole. This phenomenon was first observed by Dr. Wright, of Yale College, and was applied by him for the production of mirrors for physical apparatus. It is shown by experiments that the action in the vacuum tube is of two kinds. A silver pole was used, and near it, in front, was a sheet of mica with a hole in its centre. The vacuum was very high (P=0·00068 m.m.), and when the poles were connected with the coil, the silver being negative, electrons shot from it in all directions, and passing through the hole in the mica screen, formed a bright phosphorescent patch on the opposite side of the bulb. The action of the coil was continued for some hours, to volatilise a certain portion of the silver. On subsequent examination it was found that silver had been deposited only on the mica screen and in the immediate neighbourhood of the pole; the far end of the bulb, at the spot which had been glowing for hours from the impact of electrons, being free from silver deposit. Here then are two simultaneous actions. Electrons, or as I once called them "Radiant Matter," shot from the negative pole, and caused the glass against which they struck to glow with phosphorescent light. Simultaneously the heavy positive ions of silver, freed from their negative electrons, or under the influence of the electrical stress, likewise flew off, and were deposited in the metallic state near the pole.

An experiment was tried to discover if the ions of metal, deposited in this manner on a metal plate connected with an idle pole, in the full stream of + ions and − electrons, showed any special + or − electrification. In all cases the electrification was positive. This lends support to Mr. Strutt's view that + ions as well as − electrons will fly off from a radio-active body. Even these results, however, must not be taken as conclusive, for in a paper published in 1891 ("Electricity in Transitu: from Plenum to Vacuum," *Journ. Inst. Elect. Engin.*, vol. xx., p. 10), I showed that at a high vacuum nearly the whole of the interior of a tube through which an induction spark was passing was electrified positively, negative electrification being detected only in the immediate neighbourhood of the negative pole.

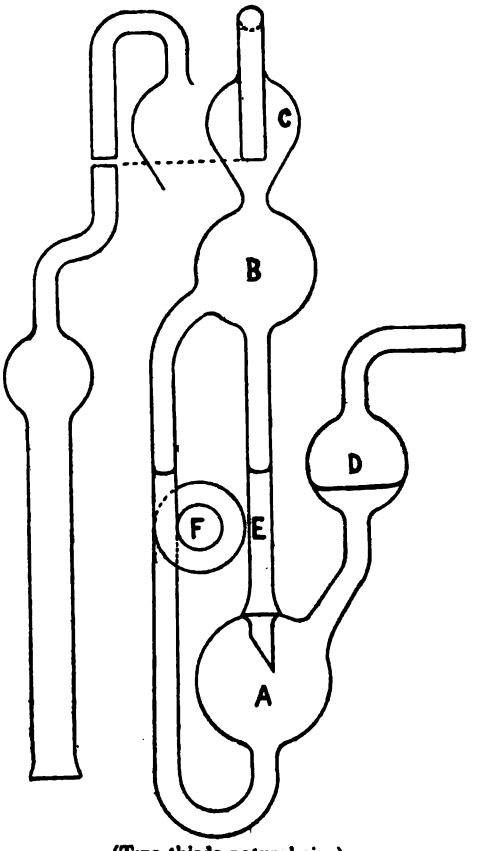
During the course of my experiments a curious circumstance was observed, which deserves record as it may elucidate some of these obscure phenomena. While the volatilisation of the silver pole is rapidly proceeding, the metal glows as if red-hot. This "red heat" is superficial only. The metal instantly assumes, or loses, the appearance of red heat the moment the current is turned on or off, showing that the high temperature does not penetrate below the surface. The volatilisation of the positive ions is confined to the surface, and the surface glow is connected with that action. If instead of silver, a good conductor of heat, I take diamond, a bad conductor, the surface layers are changed sufficiently to convert them into a form of graphite, which from its great resistance to oxidising agents, cannot be formed at a lower temperature than 3600° C.

Royal Institution.—On Tuesday next, March 11th, Professor E. B. Poulton, F.R.S., Hope Professor of Zoology in the University of Oxford, will deliver the first of a course of two lectures at the Royal Institution on "Recent Researches on Protective Resemblance, Warning Colours, and Mimicry in Insects"; and on Thursday, March 13th, Mr. E. T. Reed, of the staff of *Punch*, will commence a course of two lectures on "Caricature in and out of Parliament." The Friday Evening Discourse on Friday, March 14th, will be delivered by Professor Silvanus P. Thompson, his subject being "Magnetism in Transitu"; the succeeding discourse on March 21st will be given by Geheimrath Professor Otto N. Witt, of Berlin, on "Recent Developments in Colouring Matters" (in English). There will be no Evening Meetings on March 28th and April 4th.

A NEW DESIGN FOR POTASH BULBS.

By J. N. TERVET.

POTASH bulbs, whether made on the Liebig or on the Geissler pattern, have two disadvantages: first, the potash is not used up uniformly; secondly, crystallisation of potassium carbonate sometimes occurs and hinders the passage of the gas. To obviate these faults, I have designed bulbs, shown in the sketch, in which the current of gas itself causes the caustic potash to circulate through the whole apparatus. At the same time the mixture of gas and potash solution is so intimate that the speed of absorption is increased, and the time necessary to execute a combustion can be appreciably diminished.



(Two-thirds natural size).

It will be observed that the apparatus consists of four bulbs. The greater bulk of the potash is contained in A when not in use. The connection between the upper bulb, B, and the under bulb, A, is effected by the tube E, which is cut off obliquely at the lower extremity. In addition, connection is made by the syphon tube, F. C and D are safety bulbs.

The gas passing through D enters A and depresses the potash solution until the level reaches the oblique cut. The gas rises through E in a stream of small bubbles carrying the potash solution upward into B, from which it returns to the lower bulb, A, by means of the syphon tube, F.

The total weight of the apparatus when charged is 40 to 45 grms. The following analyses have been performed, using the apparatus:—

Dimethyl Fluoran.

0·1297 grm. substance gave 0·3829 grm. CO_2, 0·0596 grm. H_2O.

	C.	H.
$C_{22}H_{16}O_3$ requires	80·45	4·92
Found	80·51	5·15

Amyl Alcohol.

0·1200 grm. substance gave 0·2994 grm. CO_2, 0·1471 grm. H_2O.

	C.	H.
$C_5H_{12}O$ requires..	68·08	13·74
Found	68·04	13·74

Acetparabromanilide.

0·1782 grm. substance gave 0·2919 grm. CO_2, 0·0578 grm. H_2O.

	C.	H.
C_8H_8ONBr requires	44·84	3·77
Found	44·67	3·63

The apparatus may be obtained from the firm of Max Kaehler and Martini, Berlin.

East London Technical College.

A BIBLIOGRAPHY OF STEEL WORKS ANALYSIS.

PART XII.

By HARRY BREARLEY.

(Concluded from p. 103).

IRON.

III. VOLUMETRIC ESTIMATIONS (*continued*).

b. As Ferric Salts.—

1699. LANDOLT (*C. N.*, vi., 184).—Destroy free HCl with $N\bar{a}A$, add excess hypo., and titrate with I and starch.

1700. CRAFTS (*C. N.*, xxviii., 44).—Evaporate HCl solution until the crust on the surface dissolves with difficulty; titrate with hypo.

1701. OUDEMANNS (*C. N.*, xvi., 218; and xxii., 256).—To HCl solution add KCNS and titrate with hypo. Sr, Ca, Mg, Mn, Ni, Co, and Cu do not interfere.

1702. HASWELL (*J. I. S. I.*, 1883, 385).—Add soda salicylate in 1701; then hypo. till violet colour goes, and then $K_2Cr_2O_7$ till it reappears. See also OUDEMANNS and HASWELL (*C. N.*, xlvii., 210) and BRUEL (*C. N.*, xlviii., 248).

1703. NORTON (*C. N.*, lxxx., 89).—Estimation with hypo. and I. Determines some necessary relations between acidity, dilution, &c.

1704. PSZCZOLKA (*C. N.*, xlvii., 107) and PARTHIEL (*J. I. S. I.*, 1890, i., 374).—Add KI to faintly acid ferric solution, and titrate with hypo. and starch.

1705. FALIERES (*J. C. S.*, xlviii., 1011) and NIHOUL (*C. N.*, lxvii., 196).—Distil acidified Fe_2Cl_6 with KI, and titrate volatilised iodine.

1706. CARNEGIE (*C. N.*, lx., 87).—Investigates nature of reaction between Fe_2Cl_6 and KI, on which estimation of former depends.

1707. MORELLS (*J. S. C. I.*, 1884, 179).—Heat with KI, add a known amount of Hg, and shake. The free I and Hg combine and the Fe is deduced from the loss of Hg on re-weighing.

1708. WINKLER (*C. N.*, xxi., 233).—Add KCNS and then CuCl until the colour disappears and a cloud of CuCNS is formed. Co, Ni, Cu, and As do not interfere. See also GIBBS (*C. N.*, xvii., 161).

1709. WEIL (*C. N.*, xlvi., 284) and JEAN (*J. C. S.*, lxiv., ii., 492).—Add $SnCl_2$ to HCl solution of Fe_2Cl_6 until colour goes; or add $CuCl_2$ to heighten end-reaction.

1710. HEMPEL (*C. N.*, lii., 321). — Ignite with $Na_2CO_3+CaCO_3$ to eliminate organic matter, dissolve in HCl, and titrate with $SnCl_2$.

1711. MAHON (*J. S. C. I.*, 1893, 1061).—$SnCl_2$ is added to a solution containing $HgCl_2$ and a little $PtCl_3$; a faint excess precipitates a dark cloud of finely-divided Hg and Pt.

1712. MORAHT (*C. N.*, lxv., 275).—Add ferrocyanide until the sulphocyanide colour, kept in solution with ether, is dispersed.

1713. CHARPENTIER (*C. N.*, xxviii., 71).—To solution containing no free acid add KCNS and titrate with caustic alkali. A like process was suggested by ROSS (*C. N.*, xvii., 23 and 35) and adversely criticised by WRIGHT.

1714. PURGOTTI (*J. C. S.*, lxxii., ii., 77).—An acid solution of the blue oxide (Mo_3O_8) mixed with hot Fe_2O_3 solutions is quantitatively decolorised.

1715. JUETTE (*C. N.*, xvii., 63).—The amount of tartaric acid needed to retain iron (Cr, Mg, Al) in solution is proportional to the amount of the latter.

1716. CARNOT (*J. S. C. I.*, 1889, 138).—Cold acid FeO solutions are more effectually oxidised by H_2O_2 than by any other reagent.

IV. COLORIMETRIC ESTIMATIONS.

1717. DAVIES (*C. N.*, viii., 163).—Add KCNS, and compare with standard.

1718. SKEY (*C. N.*, xvi., 180 and 233).—When the mixed Fe_2Cl_6 and KCNS is treated with excess HCl, Fe is volatilised at ordinary temperatures.

1719. KRÜSS and MORAHT (*C. N.*, lxiv., 255).—The KCNS colour is due to the formation of $Fe(CNS)_3.9KCNS+4H_2O$.

1720. LUNGE (*C. N.*, lxxiii., 250).—Shakes out the KCNS colour with ether. Better to oxidise Fe with $KClO_3$ than with HNO_3.

1721. WEBER (*C. N.*, xlvii., 165).—Chlorides of the alkaline earths retard or may prevent the KCNS reaction.

1722. THOMSON (*C. N.*, li., 259).—Ag, Cu, Co, and $HgCl_2$ interfere with the KCNS colour.

1723. TATLOCK (*J. S. C. I.*, 1887, 276).—Influence of reagents, free acid, oxidisers, &c., on the KCNS colour.

1724. VERNON (*C. N.*, lxvi., 177, &c.)—Influence of temperature, dilution, acidity, &c., on the KCNS test. See also GLADSTONE (*C. N.*, lxvii., 1 and 66); ANDREWS (*C. N.*, lxx., 165); KRÜSS and MORAHT (*C. N.*, lxvi., 198)); and RIBAN (*J. S. C. I.*, 1892, 369).

1725. SABANEEFF (*J. C. S.*, liv., 757).—Colour of Am_2S solutions used.

1726. JOB (*J. C. S.*, lxxvi. ii., 51).—Fe_2S dissolved in soda pyrophosphate gives a green solution well adapted to a colorimetric estimation of Fe.

1727. CARNELLEY (*C. N.*, xxx., 257).—The blue colour formed by ferrocyanide can detect 1 part Fe in 13,000,000 of water. Process elaborated for estimation of Fe in water.

1728. WAGNER (*C. N.*, xlv., 35).—Limit of sensitiveness of some reactions for Fe and Cu.

V. MISCELLANEOUS.

1729 JULIEN (*C. N.*, xxiv., 292), BLAIR (*C. N.*, lvi., 183), and MORGAN (*J. S. C. I.*, 1894, 1023).—Schemes for elaborate analysis of iron ores.

1730. BETTEL (*C. N.*, xliii., 100).—Discusses the determination of basic cinder and oxides in iron, and reviews a number of methods proposed for this purpose. The following (not exhaustive) references are to papers dealing with the estimation of oxides or slag:—GLADEY (*J. C. S.*, lxiv., ii., 388); PARRY and MORGAN (*C. N.*, lxvii., 260); LEDEBUR (*J. S. C. I.*, 1882, 366); SCHNEIDER (*J. S. C. I.*, 1900, 693); TUCKER (*J. I. S. I.*, 1881, 205).

1731. FOEHR (*C. N.*, xlvi., 40).—Ores containing MnO_2 need prolonged boiling to expel the Cl formed, and Fe_2Cl_6 is volatile even at 50°.

1732. Fresenius (*C. N.*, xvi., 37) and Talbot (*C. N.*, lxxv., 227).—Fe_2Cl_6 is not volatilised on boiling with acids.

1733. Borntträger (*J. C. S.*, lxx., ii., 502; and lxxviii., ii., 171).—Ignited Fe_2O_3 dissolves instantly if metallic iron is also being dissolved in the HCl, or if a little MnO_2 is added.

1734. Anon. (*C. N.*, xxxviii., 11).—Ignited Fe_2O_3 can be dissolved in HCl after boiling to flocculent condition with KHO.

1735. Zirnite (*J. I. S. I.*, 1888, i., 368).—A 30 per cent NaHO solution dissolves Fe_2O_3 as $NaFeO_4$; it is re-precipitated on diluting or standing.

1736. Jeannel (*C. N.*, xvii., 286).—Properties of "neutralised" ferric chloride [$Fe_2Cl_6.9Fe_2(HO)_6$] solutions.

1737. Warren (*C. N.*, lxxii., 100).—Au and Ag were estimated in iron by making the sample (4 lbs.) the + pole of an acid battery. The C residue and a little Fe were mixed with litharge, cupelled, &c.

1738. Warren (*C. N.*, lxxv., 91).—Samples which have been in contact with a borax flux contain B up to 2 per cent.

1739. Wiborgh (*C. N.*, lxxviii., 4).—Determines the reducibility of Fe ores. The reduced Fe is estimated by measuring the H evolved on treating with acids.

1740. Meunier (*C. N.*, xix., 5).—Immediate analysis of meteoric iron.

1741. Ledebur (*J. I. S. I.*, 1894, ii., 483).—Uses the ether method to separate iron from Cu, Sb, As, Pb, Cr, V, Mn, Ni, Co, and Ti existing in steel.

1742. J. T. (*C. N.*, lxxix., 157).—Insoluble specular ores are mixed with asbestos filter-paper and ignited; they are then soluble in HCl.

1743. Bechamp and Saintpierre (*C. N.*, iv., 316).—Metallic Pt reduces HCl solutions of Fe_2Cl_6, and is itself dissolved thereby.

1744 Dudley (*C. N.*, lxxv., 257). — "Some Present Possibilities in the Analysis of Iron and Steel."

ON THE AGRICULTURAL VALUE OF THE NITROGEN OF THE BLACK MATTER IN PYRENEAN PHOSPHATES.

By JULES JOFFRE.

It has long been known that many organic materials, although containing nitrogen, have not the slightest beneficial action on vegetation. Boussingault made the remark that coal, though containing nitrogen in the organic state, did not cause, in spite of that, any chemical action favourable to the growth of plants.

In recent years considerable deposits of phosphates have been found in the Pyrenees containing a black nitrogenised organic substance. It seemed to me to be of interest to ascertain what is the agricultural value of this substance.

For this purpose I have carried out two series of experiments laid down on the same lines as those of which I have already published a description, taking as bases of comparison, on the one hand, nitrate of soda, sulphate of ammonium, and dried blood; and on the other hand, coal. In each pot I put the same amount of nitrogen in these different forms. The sample of Pyrenean phosphate on which I experimented contained 0·14 per cent of nitrogen. Naturally, I added some insoluble phosphate to equalise the relatively high proportion that the Pyrenean phosphate contained. Further, in all the pots I put sufficient quantities of potash, lime, magnesia, and soluble phosphoric acid.

Under these conditions the nitrated organic matter of the Pyrenean phosphate behaved in the same manner as the coal. It did not cause the slightest augmentation of weight of the plants submitted to its action. On the other hand, the nitrate of soda, the sulphate of ammonium, and the dried blood produced a considerable increase of the crop.

The result of these experiments does not in any way diminish the importance and great future which M. David Levat attributes to the deposits of this Pyrenean phosphate; the more so as the amount of nitrogen contained always being small could not have much influence on the final value of this material. Still it appeared to me to be of use to publish my experiments so that agriculturalists might not be mistaken in the value of the nitrogen present in this material.—*Bull. Soc. Chim.*, Series 3, vol. xxv., No. 22.

ON THE ESTIMATION OF TIN BY LENSSEN'S PROCESS.

By J. A. MULLER.

The volumetric estimation of tin by means of an iodine solution is frequently used in laboratories on account of the rapidity with which the operation can be performed and the sharpness of the end of the reaction of the iodine on the tin in alkaline stannous combination.

This estimation—as far as we can judge from the figures quoted later on—gives, as a rule, however, rather too low results, even when taking every precaution to prevent the oxidation of the stannous solutions, as much during the solution of the metallic mass in hydrochloric acid as during the titration.

With the object of checking Lenssen's method I commenced by preparing pure iodine by submitting commercial doubly-sublimed iodine to five more successive sublimations. To obtain any determined weight of this metalloid a quantity, weighed approximately, was rapidly transferred to a ground glass stoppered weighing bottle by means of a funnel; the stopper should be slightly smeared with vaseline, and the whole previously tared; the stopper must be inserted immediately, and the bottle placed on the balance. The iodine was then dissolved in the same bottle, taking care not to lose any of the vapour, by means of a concentrated solution of pure iodide of potassium, obtained by evaporating a perfectly clear alcoholic solution of this salt *in vacuo*. The solution of iodine was then diluted to a known volume with cold, boiled, distilled water.

The tin used for these experiments gave me the following centesimal results by gravimetric estimation:—

Sn	99·76
Fe	0·13
Pb	0 05
	99·94

For the purpose of titrating, this metal was dissolved in pure, concentrated hydrochloric acid (10 c.c.). The solution was effected in a test-tube kept in an inclined position, and fitted with an indiarubber stopper through which passed a curved tube, the end of which was plunged into a small flask containing a little water. Only a very moderate heat is required, and as soon as the solution is complete the contents of the test-tube are run off, and the tube rinsed—with cold, boiled, distilled water—into a large conical beaker full of carbonic anhydride. We then add 80 c.c. of a 10 per cent solution of Seignette salt prepared with boiled water; then, at several intervals, enough carbonate of soda, in powder, to make the solution distinctly alkaline, taking good care to incline the beaker so as to prevent any loss by projections. Finally, into the limpid solution we slowly run, by means of a pipette and with constant stirring, a volume of iodine solution, almost sufficient to produce total oxidation; then, after the addition of a little starch emulsion, the titration is finished with the help of a burette.

A blank experiment, made under the same conditions, gave a solution which one drop of iodine liquor coloured blue; on the other hand, by adding to 80 c.c. of the solution of Seignette salt 5 c.c. of a solution of stannous chloride titrating 1·3 c.c. of iodine, and then titrating after about an hour, after the addition of carbonate of soda, I re-obtained an iodine value of 1·28 c.c.; the medium with which the stannous chloride was mixed, after the solution of the tin, was thus neither reducing nor oxidising.

All the weighings and gauging of the vessels used for measuring the volumes were carried out by means of the same certified weights.

The following are the results obtained :—

Weight of tin dissolved.	Volume of iodine liquor used.	Decinormal coefficient of the liquor.*	Corresponding weight of tin.	Weight of tin.
Grm.	C.c.		Grm.	Per cent.
0·5286	88·66	0·9930	0·5216	98·68
0·4609	77·23	0·9940	0·4549	98·69
0·4131	69·23	0·9940	0·4077	98·70
0·4529	75·07	1·0108	0·4496	99·27
0·4054	67·24	1·0108	0·4027	99·34
0·6009	99·36	1·0108	0·5951	99·03
0·6329	104·64	1·0108	0·6267	99·02
1·6304	99·77	1·0595	0·6263	99·35

* In these experiments we took the figure 126·85 as the atomic weight of iodine, and 118·5 as that of tin. One c.c. of decinormal iodine liquor thus corresponded to 0·005925 grm. of tin.

As the tin used might have contained a little tungsten, which is difficult to detect directly, I treated a certain quantity with nitric acid, then after washing and drying I reduced the residue by means of cyanide of potassium. By operating with the pure tin thus obtained, in the same way as with the tin containing 99·76 Sn and 0·13 Fe used in the preceding experiments, the titration gave 99·31 and 99·24 instead of 100.—*Bull. Soc. Chim.*, Series 3, vol. xxv., No. 23.

ON PYRITE AND MARCASITE.*

By H. N. STOKES.

(Continued from p. 93).

IX. Form of Occurrence of Copper in Copper-bearing Pyrites.

The problem of determining whether a given specimen of cupriferous pyrite contains its copper in the form of chalcopyrite or chalcocite was suggested by a geologist, and the following shows that such determination is possible when marcasite or other sulphides are not present in considerable quantities.

Oxidation Experiments.

Mixtures of pyrite with chalcopyrite, chalcocite, and bornite were prepared, each containing 3 per cent copper. For chalcopyrite and chalcocite these mixtures have the following composition :—

Pyrite	91·33	or	Cu	3·00
Chalcopyrite ..	8·67		Fe	45·20
			S	51·80
Pyrite	96·24	or	Cu	3·00
Chalcocite ..	3·76		Fe	44·85
			S	52·15

The bornite mixture has an intermediate composition. It would be scarcely possible to distinguish these by ordinary analytical methods, especially in the presence of small amounts of impurity. The values of ρ were found to be—

Pyrite-chalcopyrite	62·7
Pyrite-chalcocite	75·9
Pyrite-bornite..	76 4

* From the *Bulletin of the U.S. Geological Survey*, No. 186.

It appears that the oxidation method does not admit of distinguishing chalcocite and bornite with certainty in a pyrite with 3 per cent copper, but that these may be readily distinguished from chalcopyrite even when the copper is considerably less than 3 per cent. It can also be determined with some probability whether a mixture of chalcopyrite with the other sulphides of copper is present. In the present case the figures given by chalcocite or bornite and chalcopyrite differ by more than thirteen times the probable error of a determination.

This method will probably be of value in synthetic studies of copper ores. It is thus made possible, for example, to ascertain whether the first action of a cupric salt on pyrite is to produce chalcopyrite alone or a mixture of this with the other sulphides, without having to wait for the slow process of crystallisation to produce the minerals in a form in which they can be recognised under the microscope. By employing fine powders the rapidity of the change will be vastly increased, while the net results, according to the mass law, will be the same.

Detection of Chalcopyrite in Pyrite or Marcasite and in Rocks.

Chalcopyrite mixed with pyrite or marcasite may be readily detected, if not in too small amount or too finely divided, by exposing the sample to bromine vapour for half a minute, and then to hydrogen sulphide gas; the chalcopyrite is blackened, while the iron sulphides remain bright. Particles of chalcopyrite which would otherwise be overlooked are thus easily detected and their outlines sharply defined. I have used the same procedure to detect and establish the nature of minute grains of chalcopyrite enclosed in rocks.

(To be continued).

PROCEEDINGS OF SOCIETIES.

PHYSICAL SOCIETY.

Ordinary Meeting, February 28th, 1902.

Prof. S. P. Thompson, President, in the Chair.

A paper on "*Focal Lines and Anchor-ring Wave-fronts*" was read by Prof. J. D. Everett.

When a small cone of rays is obliquely incident on a spherical reflecting or refracting surface, the rays after reflection or refraction no longer compose a true cone. Instead of meeting in a point they form a narrow neck, and this neck is flattened in two places, called the *two focal points*, the planes of flattening being at right angles to each other. Optical writers give the name *focal lines* to the sections of the pencil made at the focal points by planes perpendicular to the axis of the pencil; but it would be more appropriate to give the name to the sections which most nearly resemble lines, whatever angle they make with the axis of the pencil. Attention is drawn in the present paper to the case in which the wave-front in one of its positions is a tore (or anchor-ring). Even when dealing with wide angled pencils there are then two well-defined focal lines, the primary focal line being what may be called the circular axis of the tore, and the secondary a portion of the line about which the generating circle turns to form the anchor-ring. Toric wave-fronts can be produced by reflection from a mirror made by allowing an ellipse or portion of an ellipse to revolve completely round an ordinate erected at one focus, and employing it to reflect rays diverging from a small source at the other focus. The primary line is always real; the secondary is real or virtual according to the position of the area of incidence of the pencil.

Mr. R. J. Sowter pointed out that when a thin oblique pencil, or small cone of rays, is reflected or refracted by a

spherical surface, the pencil rays form a narrow neck, and that this neck is flattened in two places called the *focal lines* and not the *focal points*; and that the focal lines cut the principal ray or axis of the pencil in the focal points. In the case of thin pencils the focal lines are elementary, and may be considered as straight lines, and an infinite number of planes can pass through them or contain them. This leads to the mutually rectangular primary and secondary planes of the pencil being selected. But if the elementary focal line is considered curved, as it is in general, only one containing plane can be chosen, namely, the plane of the curve. Real or physical focal lines are not in planes related with the focal points as suggested by Prof. Everett. It was also pointed out that the criterion for anchor-ring or toric waves is that the focal lines are respectively a circle and a straight line perpendicular to the plane of the circle and lying in its axis, and that a toric wave-front could be formed by the refraction of a hollow cone of light at an annular ring on a spherical surface. The wave-front in the refracting medium is then toric; that is, it is a circular trough which is part of an anchor-ring. The primary focal line is a circle and the secondary a straight line of limited length.

The CHAIRMAN said that optical writers often used the term focal lines to denote the sections of the wave surface made by two mutually perpendicular planes through the focal points.

A paper entitled "*Contributions to the Theory of the Resolving Power of Objectives*" was read by Prof. EVERETT.

The practical limit to the resolving power of objectives depends upon the blurring due to diffraction. Observations on double stars for the purpose of investigating the separating power of telescopes have been made by Dawes, who arrived at the conclusion that the angular distance between the two components, when they are nearly equal in magnitude and are just separated, is given by the formula, 4·56 seconds divided by the diameter of the objective in inches. Foucault also investigated the matter experimentally, and in 1830 Airy calculated the brightness at various points of the spot and rings which constitute the image of a point source formed by an objective. If the extreme difference of optical path for disturbances coming from different points of a concave wave-front to a point at lateral distance b from the geometric focus is made equal to the wave-length of light, a value for b is obtained which represents with fair accuracy the limit of separation as determined by experiment. The formula agrees with that of Dawes if $\lambda = 0{\cdot}56$ micron., whereas the wave-length of the brightest ray is usually taken as 0·55 micron. In the case of microscopes the author has supposed that the formula for the minimum distance b still holds good, and combining this equation with the sine condition applicable to optical systems giving sharp flat images, he has deduced the expression which has been extensively used for the distance between lines or points which can be barely separated. Microscopic test objects are not self-luminous, like double stars, but are viewed by transmitted light. If no condensing arrangement is employed, the pencil of light sent by a point of the object to the objective consists, in effect, of rays from different sources. The result of this is that the image of the point is larger and more blurred. The cure for this evil is furnished by employing a condenser of high quality, to throw upon the part of the object under examination a very sharp image of the source of illumination. Each point of the object thus gets its light from its own special point of the source; the object, therefore, acts as if it were self-luminous, and the power of the instrument is increased. The benefit derived from sharp focussing on the object explains the advantage of using an achromatic condenser, and not, as formerly recommended by Abbe, one which is not achromatic. Another advantage of sharp focussing by the condenser is that there can be no interference of the light from different parts of the object. The author then gives an explanation of the advantage of oblique illumination, and arrives at the view of microscopists that the obliquity of illumination should be rather less than the obliquity of the extreme rays of the incident pencil. The paper concludes with Hockin's proof of the sine-condition.

The CHAIRMAN expressed his interest in the paper, and referred to the rational reason given for the use of an achromatic condenser.

A paper on "*The Absorption, Dispersion, and Surface Colour of Selenium*," by Prof. R. W. WOOD, was read by the SECRETARY.

The dispersion of selenium has been investigated by means of prisms made in the same manner as the cyanine prisms already described by the author. The substance is much more transparent than cyanine, and prism angles of four or five degrees can be employed. Determinations were made with three selected prisms down to wave-length 61; below this the interferometer method was employed. Uniform films of selenium were obtained on plates of plane parallel glass, by means of a flat selenium cathode in a high vacuum, and the displacements of the interference fringes by the introduction of the films were measured for lights of different known wave-lengths. Wedge-shaped films were then employed which allowed the displacement for any wave-length to be measured for the maximum thickness capable of transmitting the light. An advantage of the wedge-shaped films is that the fringes are curved, and the displaced fringe can be easily identified with the undisplaced. The refractive indices obtained in the red by prisms were used as a basis for the calculation of the indices in the rest of the spectrum from the interferometer measurements. Determinations were made in this way down to wave-length 40, beyond which it was impossible to go owing to the powerful absorption. A curve has been plotted showing the relation between refractive index and wave-length. It has a maximum at wave-length 50 (0·00005 c.m.) where the refractive index is 3·13. An examination of the light transmitted by a thin film showed that there was no return of transparency in the ultra-violet, at least down to wave-length 28. Photometric measurements were made of the transmitted light, both visible and ultra-violet, and a curve has been drawn with wave-lengths as abscissæ, and extinction coefficients as ordinates. It is proved that the extinction coefficient increases continuously with decrease in wave-length as far as wave-length 22, where the coefficient has as high a value as in the case of metals. The author concludes that the absorption is due, not to a single band, but to a series of overlapping bands. The object of this work was to determine whether there was a return to partial transparency in the ultra-violet region. This question appears to be answered in the negative although a possible turning point in the curve might be masked by the reflection coefficient of selenium. The high value of the extinction coefficient in the ultra-violet led the author to look for traces of selective reflection in this region. The light of an arc lamp was reflected successively from six surfaces of selenium, and the image of the crater after the sixth reflection, although faint, was without colour or excess of ultra-violet light. If the data obtained in the paper for refractive index and extinction are used in the formula for reflection from an absorbing medium a result is arrived at which indicates that the reflection increases rapidly with decreasing wave-length. As multiple reflections from selenium surfaces give no trace of colour, errors must exist in either the refraction or the extinction curve. The author suggests that in the case of films of thickness less than the wave-length of light, the displacement of the interference fringes does not give a measure of the refractive index.

The CHAIRMAN exhibited some Tellurium Mirrors made in the same way as the selenium ones used by Prof. Wood.

The Society then adjourned until March 14th.

MANCHESTER LITERARY AND PHILOSOPHICAL SOCIETY.

Ordinary Meeting, February 18, 1902.

Mr. Charles Bailey, President, in the Chair.

Mr. R. L. Taylor read a paper "*On a Modification of Rose's Method of Separating Cobalt and Nickel.*"

In the original process described by Rose and improved by T. H. Henry, barium carbonate and chlorine (or bromine) were added to rather strongly acid dilute solutions of the two metals, and allowed to stand, with frequent shaking, for from twelve to eighteen hours. The cobalt was precipitated as sesquioxide, while the nickel remained in solution. Mr. Taylor finds that if a neutral solution is used the precipitation of the cobalt is complete in a few minutes, and that excellent quantitative results can be obtained. The retardation of the reaction which occurs when the solution is (as Rose and Henry used it) strongly acid at the outset, the author shows, is due to the free carbonic acid which is produced in the solution when the carbonate is added to the acid liquid. A similar retardation occurs in a neutral solution if carbon dioxide is first bubbled through it or if soda-water is added to it. Mr. Taylor recommends the process for the separation and detection of cobalt and nickel in the ordinary process of qualitative analysis. Either barium or calcium carbonate may be used (dry precipitated, as usually sold, will do) with bromine-water, and if no free acid is present at the outset the cobalt is all precipitated in five minutes. On filtering, nickel can be readily detected in the filtrate by adding a little ammonia and ammonium sulphide. If there is any free acid present at the outset, it must either be boiled away or neutralised before adding the carbonate. Sodium carbonate may be used for neutralising, but then the free carbonic acid must all be boiled away, and the liquid cooled before adding the carbonate and bromine water.

Mr. D. L. Chapman described some experiments which have been carried out, in conjunction with Mr. F. A. Lidbury, principally for the purpose of discovering whether Faraday's law may be considered as applying to gases.

The electric discharge was passed through water vapour, and the separation of oxygen and hydrogen which took place was found to be from two to three times as great as that which occurred in a voltameter placed in the same circuit. The results are therefore inconsistent with the view that the phenomenon is essentially electrolytic.

NOTICES OF BOOKS.

Enzymes and their Applications. By Jean Effront. English Translation by Samuel C. Prescott. Vol. I., "The Enzymes of the Carbohydrates; the Oxidases." New York: John Wiley and Sons. London: Chapman and Hall, Ltd. 1902. Pp. xii.-322. 8vo.

The number of treatises on soluble ferments in the English language is comparatively small, and makes this translation of Professor Effront's standard volume the more highly appreciated by students of bio-chemistry. In its English dress the original has been presented in the form in which it came from the pen of the author without additions or changes; it is actually a summary of the course given at the Institute of Fermentations of the new University of Brussels, and is designed both for students of pure science and for those particularly occupied in fermentation studies.

In successive chapters the author treats of the general properties of enzymes, of the action of diastases, of the individuality and classification of enzymes, of sucrase, amylases, maltase and its industrial applications, zymase, and the oxidases, as well as of panary fermentation and the fermentation of molasses.

The classification of soluble ferments adopted by the French author is based on their chemical behaviour, and is as follows:—I. The soluble hydrating ferments. II. The soluble oxidising ferments. III. The ferments causing molecular decomposition. Class I. embraces the soluble ferments of (*a*) carbohydrates (the subject of the present volume), (*b*) glucosides, (*c*) fatty substances, (*d*) proteins, (*e*) urea. Classes II. and III. are more briefly treated owing to less extensive knowledge. In discussing nomenclature M. Effront expresses regret that the rational system suggested by Duclaux has not been more universally adopted, for although it was not all that could be desired, it would have prevented confusion. He decides to use the names generally met in the literature of the subject.

In writing of the industrial applications of the soluble ferments the influence of French methods is naturally prominent; this appears in the chapter on panary fermentation, which, though scientifically accurate, does not entirely harmonise with American practice, nor with English processes that have not been affected by the customs of their friends across the Channel.

A valuable feature of the book is the bibliography annexed to each chapter, which, however, refers to journal-literature not later than 1898; in a second edition the translator might well extend this bibliography to a more recent date.

There is a rather brief index. H.C.B.

Elementary Chemical Theory. By G. H. Martin, M.A., F.C.S., Assistant Master at Bradford Grammar School. New Edition, Revised. London: Rivingtons. 1902. Pp. 24.

As its title indicates, this book is purely elementary, and is evidently intended for the use of young beginners. It begins with a definition of matter, which is described as anything having *weight*. Dalton's atomic theory is next explained, followed by Gay-Lussac's and Avogadro's laws. Next we come to symbols, formulæ, and molecular weights and valency, all taken seriatim. Part II. describes the different kinds of elements—metals and non-metals—bases and acids, nomenclature and equivalent, or combining weights, &c. The book appears to be well adapted to the needs of school-boys; it touches not only on elementary chemistry, but also on elementary physics, both of which are treated in a clear and concise manner; it is interleaved with blank pages to enable the student to make his own notes and observations, as his fancy takes him, and is indexed.

Year-Book of the United States Department of Agriculture, 1900. Washington: Government Printing Office. 1901. Pp. 880.

This volume contains, besides the report of the secretary and the appendix, thirty-one articles; that is, five more than last year. With one exception every article was prepared by an employé of the department, and each division of original work is represented by one or more articles, each one covering some important line of work carried on. Thus the year-book for 1900 differs from some of its predecessors, which were devoted more especially to a review of the work of the several departments.

The illustrations, which are very well executed, comprise eighty-seven plates, nine of which are coloured, and eighty-eight figures in the text.

The work done, in a long life devoted to agriculture, horticulture, and kindred interests by the late Mr. William Saunders, is the subject of a brief notice by the editor, which fully justifies the prominent place given to his memory in this edition.

In the appendix, which it is desired to make an indispensable book of reference to the farmer, the agricultural

directory feature, introduced in 1899, has been continued; it has also been revised and corrected up to March 1st, 1901.

The statistics of farm crops, supplied as usual by the Statistician of the Department, have been re-arranged so as to present together the facts and figures relating to each crop, thus affording one complete view of results for a particular crop for the year. This change will be found, it is believed, especially convenient to farmers who have occasion to consult these figures in planning their future operations.

Cyanide Practice. By Alfred James, Mining and Metallurgical Engineer, Member of the Institution of Mining and Metallurgy, F.R.G.S., F.C.S., &c., Late Technical Manager and Special Expert in South Africa, New Zealand, Australia, United States, and elsewhere of the Company introducing the Cyanide Process. London: E. and F. N. Spon, Limited. New York: *Engineering and Mining Journal* (Incorporated). 1902. Pp. 164.

While the same general principles govern the employment of cyanide for the extraction of gold from its ores, it must be remembered that modifications are numerous, and that new details of practical working are constantly coming into being. Possibly the most remarkable tribute to the efficiency of the cyanide process is the practical relegation to the back-ground of the numerous processes which formerly reappeared so persistently under new names, and in the hands of different inventors, all claiming the highest possible extractions at a minimum of cost.

A great deal of technical skill is necessary for the proper working of the cyanide process, there being many reasons why the extractions may be poor; for instance, soluble sulphides may be formed, the gold may be too coarse or in the form of telluride; again, base metals may be present, and these enter into combination with the cyanide and consume it. For the treatment of sulphide ores bromo-cyanide may be added, or the metallic sulphides may be eliminated by fine grinding, and the addition of a freshly precipitated lead salt, such as the chloride or carbonate, to decompose any soluble sulphide as soon as it is formed. In all cases of heavy consumption of cyanide it is as well to analyse the solution, and to determine the amount of cyanide accounted for by the metals in solution. Ores high in silver will naturally consume a good deal of cyanide. The author has met with gold-silver ores, not concentrates, for which 6 lbs. of cyanide have been required per ton of ore treated, merely to account for the silver in solution. Having found the amount of metals in solution, if the copper be multiplied by 3, the iron by 7, the silver by 1·2, and the gold by 0·7, the consumption due to each metal can be determined; if there is still any considerable balance, it may be due to the presence of acid salts in the ore, or in the water used.

The most important recent improvement in the cyanide process is the introduction of bromo-cyanide. The addition of bromo-cyanide to the ordinary cyanide solution in the proportion of not more than one part of the former to four of the latter, considerably accelerates the rate of solution of the gold, though it may not affect the final percentage of extraction; its addition, however, generally causes a greater consumption of cyanogen. Bromo-cyanide *per se*, that is, apart from its reactions in the presence of cyanide of potassium, can scarcely be claimed as a solvent of the precious metals. Indeed, as the author points out, it is remarkably stable until added to the cyanide solution, but if in addition gold is also present a reaction will take place as follows:—

$$3KCy + BrCy + Au_2 = 2KCy + Cy_2 + Au_2 + KBr = 2KAuCy_2 + KBr,$$

and it is undoubtedly due to this reaction that the extensive use of bromo-cyanide is due, whether as an accelerator or as an aid to actual solution. The author also shows that the presence of air is not only unnecessary for the solution of gold in the presence of bromo-cyanide, but that its addition causes no improvement in the extraction; on the other hand, when ferri-cyanide is being used the improvement shown by the addition of air is most marked.

We strongly recommend this book to the notice of any of our readers who are interested in gold extraction; it is exceedingly well written, and quite up-to-date; it is adorned with twenty-five illustrations, most of them being photographs of actual cyanide plants in operation in various parts of the world. The table of contents is full and comprehensive, but there is unfortunately no index. Possibly this omission may be remedied in a future edition.

CORRESPONDENCE.

CHEMICAL NOMENCLATURE.

To the Editor of the Chemical News.

Sir,—As I see two letters in your issue of December 6th last on the subject of chemical nomenclature sent from America I feel encouraged to write to you, as, although the idea of doing so on various matters has often occurred to me, I have felt myself so far away that it appeared useless.

My greatest difficulty in memory is in the names of persons, and therefore I quite agree with your correspondent E. Booth, that it would be advisable to drop the names of propounders of theories or laws and state instead what they refer to. I have seen examination papers in which such names have been used, in which, although I could state all the laws, &c., I could not connect them with the personal name, and so would probably have been plucked so far as these were concerned. Such questions are not an examination in pure chemistry, but to a large degree in verbal memory.

There is, however, even a worse evil continually growing, especially in communications from public analysts, of indicating processes, reagents, apparatus, &c., by the names of the proposers; thus tending to convert the chemistry of the analyst into the jargon of a trade line, the dog Latin, and trade marks of the pharmacist. I have a small French book, "Répertoire des Reactifs Speciaux," by F. Jean and C. Mercier, giving a description of some hundreds of these, but one finds many more, and they are growing every day. It is quite as easy to say of the determination of nitrogen, the soda-lime process, as Varrentrapp and Will's process, or sulphurous acid process, as Kjeldahl's process. It may be objected that a little more space would be occupied, but it is found that the same writers do not save words. Thus I noticed to-day that one writer suggested doing something "by means of" a platinum wire, when "with" would have have been better—nine letters used instead of four.

Another expression to be condemned is the continual use of the words estimate or estimation, in connection with chemical work; they being used as if synonymous with determined or determination, conveying to outsiders an idea of laxity and approximate guess-work; as an estimation is only a probable guess at a number formed from imperfect data, whilst a determination is the result of actual weighing, measuring, or counting; subject, of course, in each case to errors arising from personal equation, &c., which are, or should be, reduced to the lowest possible limits. The Chancellor of the Exchequer estimates the revenue and expenditure of the coming year, but the determinations only come at the end of the time, and may be much over or under the estimates. An architect estimates the cost of a house, various builders do the same, and are prepared to contract for its erection, but these generally differ widely; the determination is found when the house is finished and the bill is presented. Some years ago I

had a case in point where a quartz reef was worked for gold, the average of all assays of the stone not differing much, being 3½ ozs. per ton; but the gold recovered in the battery, and from the pyrites saved, was only 1½ ozs. I suggested that the gold was lost in the pyrites, but was told all was saved. The directors, under my advice, got samples taken systematically of the sand from the battery containing the pyrites. The battery manager *estimated* that the stone contained 2½ per cent of pyrites, and he saved that quantity; the *determination* of the pyrites gave 23 per cent. His estimate was based on the yield he had got from previous similar stone.—I am, &c.,

WILL. A. DIXON, F.I.C., F.C.S.

Chemical and Assay Laboratory,
97, Pitt Street, Sydney.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxiv., No. 6, February 10, 1902.

Crystallisation of Chromium Sesquioxide.—Alfred Ditte.—When potassium bichromate is heated to bright redness with common salt, and the mass when cool is treated with water, chromium sesquioxide remains crystallised in thin brilliant oily plates. The author experiments on the reason of this phenomenon and finds (1) that the crystallisation of the chromium oxide is not due to its solubility in the fused alkaline chloride, and (2) that the sodium salt is apparently very favourable to crystallisation, whilst the potassium salt seems to hinder it. Further experiments show that the phenomenon is really due to the formation of an oxychloride of chromium, CrO_2Cl_2, and its combination with the halogen sodium salts.

Condensation of the True Acetylenic Carbides with Aldehydes; Synthesis of Secondary Alcohols containing an Acetylenic Function.—Ch. Moureu and H. Desmots.—The authors recently discovered, whilst examining the condensation of the true acetylenic carbides with formic aldehyde, a very simple method of synthesising primary alcohols of acetylenic function—

$$R-C\equiv C-CH_2OH.$$

An extension of this method to other aldehydes led to the synthesis of secondary acetylenic alcohols—

$$R-C\equiv C-CHOH-R',$$

a group of compounds totally unknown up to the present. The experiments were performed on the one hand with four carbides—

Butylacetylene .. $CH_3-(CH_2)_3-C\equiv CH$,
Œnanthylidene .. $CH_3-(CH_2)_4-C\equiv CH$,
Caprylidene .. $CH_3-(CH_2)_5-C\equiv CH$,
Phenylacetylene . $C_6H_5-\equiv CH$,

and, on the other hand, with six aldehydes—

Acetaldehyde, CH_3-CHO,
Chloral, CCl_3-CHO,
Valeral, C_4H_9-CHO,
Œnanthol, $CH_3-(CH_2)_5-CHO$,
Benzylic aldehyde, C_6H_5-CHO,
Furfurol, C_4H_3O-CHO.

In all cases condensation took place by the solution of the sodium carbide in an ethereal solution of the aldehyde. By this means ten acetylenic alcohols were isolated, all new compounds.

Iodine Phenols.—P. Brenans.—During the action of iodine on sodium phenate, the latter being in suspension in carbon disulphide, M. Schall obtained with orthoiodophenol a triiodophenol and a diiodophenol, to which substances he gave no definite constitution. The author's present research establishes that the triiodophenol derivative is the compound $OH-C_6H_2-I_3(1,2,4,6)$, and the diiodophenol derivative is $OH-C_6H_3-I_2(1.2.6)$.

Action of Crystallised Arsenic Acid on Pinene.—P. Genvresse.—During the action of arsenic acid on pinene the author obtained a body having a different odour from that of essence of terebenthine, a certain amount of terpinene, in addition to a little cymene and terpinol, being formed.

MISCELLANEOUS.

Dr. Morris Travers's Book on "The Study of Gases."—In our review of this book in the CHEMICAL NEWS for February 28th, we remarked on the absence of the plates of spectra of certain gases. We understand from Messrs. Macmillan, the publishers of the book, that the omission of these plates was due to an accidental oversight on the part of the binder, and was confined to a few only of the early copies which were sent to the Press for purposes of review. An examination of the plates confirms the opinion we expressed that they would be of the greatest value to all engaged in the study of the newly discovered inert gases of the atmosphere.

Royal Institution.—A General Monthly Meeting of the Members of the Royal Institution was held on the 3rd inst., Sir James Crichton-Browne, Treasurer and Vice-President, in the Chair. It was announced that his Royal Highness the Prince of Wales had graciously consented to become Vice-Patron of the Institution. Sir William Agnew, Bart., Mr. Hilder Daw, Mr. J. F. W. Deacon, Mrs. Deacon, Mr. A. St. John Clerke, Miss Agnes M. Clerke, Mr. E. Figgess, Mr. M. Fitzmaurice, Mr. F. M. Guedalla, Dr. R. Harold, Mr. F. Legge, Dr. H. Lewkowitsch, Miss A. H. Little, Mr. W. F. Preedy, Dr. A. M. Robson, Mr. M. M. Samuel, Mr. F. A. Smith, Mr. J. J. Torre, Major-General James Waterhouse, Mr. Philip Watts, F.R.S., and Mr. C. Tweedale were elected Members. The special thanks of the Members were returned to "An Old Member" for a donation of £50 to the Fund for the Promotion of Experimental Research at Low Temperatures.

The Iodisation of the Mixed Hydrocarbides.—A. Erdinger and P. Goldberg.—Following up their previous researches, the authors have endeavoured to utilise for the purpose of iodising certain substances the decomposition undergone by iodide of sulphur under the influence of certain reagents; as is well known iodide of sulphur is decomposed when heated with HNO_3 (D = 1·2), and they have attempted to utilise this reaction. Their first object was to examine this reaction, in such cases where the known methods of ioduration are not applicable, or only give bad results; such is particularly the case with the mixed aromatic hydrocarbides. Among other results the authors have found that their method renders possible the direct introduction of iodine into the radical of the aromatic compounds without attacking the lateral chain, so that mono-iodides can be obtained. The method has also been applied to the preparation of iodobenzene, although this compound has already been obtained, with excellent returns, by Neumann and Istrati, by iodising in the presence of concentrated sulphuric acid. For iodobenzene, for example, they heat on the water-bath, with a vertical condenser, 10 grms. of benzene, 20 grms. of powdered iodide of sulphur, and 120 c.c. of HNO_3 (D = 1·34) for two or three hours. The ortho- and para-iodotoluenes were prepared in an analogous manner, using benzene as the solvent; the ortho-iodoxylenes, mesitylene, durol, pentamethylbenzene, cymene, and naphthalene were submitted to the same process of ioduration, and as a rule good results were obtained.—*Berichte*, vol. xxxiii., p. 2875.

MEETINGS FOR THE WEEK.

MONDAY, 10th.—Society of Arts, 8. (Cantor Lectures). "Photography applied to Illustration and Printing," by J. D. Geddes, F.R.P.S.

—— Society of Chemical Industry, 8. "Birmingham Sewage and its Treatment," by F. R. O'Shaughnessy, A.R.C.Sc (Lond.), A.I.C., &c. "Remarks on the Technical Examination of Glue," by E. G. Clayton, F.I.C., F.C.S.

TUESDAY, 11th.—Royal Institution, 3. "Recent Researches on Protective Resemblance, Warning Colours, and Mimicry in Insects," by Prof. E. B. Poulton, M.A., Hon. LL.D., D.Sc., F.R.S.

WEDNESDAY, 12th.—Society of Arts, 8. "The Utility of Alkaline Phosphatic Manures," by John Hughes, F.I.C.

THURSDAY, 13th.—Royal Institution, 3. "Caricature in and out of Parliament," by E. T. Reed.

—— Society of Arts, 4.30. "The Indian Famine of 1899, and the Measures taken to meet it," by Thomas William Holderness, C.S.I.

FRIDAY, 14th.—Royal Institution, 9. "Magnetism in Transitu," by Prof. Silvanus P. Thompson, B.A., D.Sc., F.R.S.

—— Physical, 5.

SATURDAY, 15th.—Royal Institution, 3. "Some Electrical Developments," by Lord Rayleigh, F.R.S., &c.

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Chemistry Applied to Manufactures and Art. 8 vols., 1880. 25s.
Spon's Encyclopædia of the Industrial Arts. 2 vols., 1882, half morocco. 55s.
Percy's Metallurgy. Refractory Materials and Fuel, Gold and Silver, Iron and Steel, Lead, Copper, &c. 5 vols. 1861-80. £4 7s. 6d.
Remsen's Text-Book of Inorganic Chemistry. 1889. 8s. 6d.
Roscoe and Schorlemmer's Chemistry, complete. 9 vols. 1877-92. £4.
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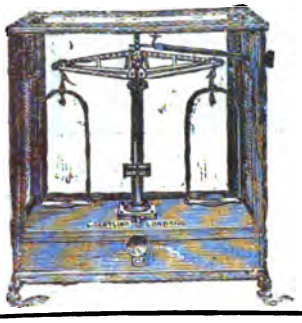

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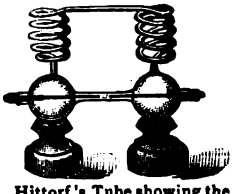

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THE CHEMICAL NEWS.

Vol. LXXXV., No. 2207.

THE ESTIMATION OF CARBON IN STEEL BY DIRECT COMBUSTION.

By RUDOLF L. LEFFLER, F.C.S.

Many people entertain the opinion expressed in Arnold's "Steel Works Analysis" regarding the estimation of carbon in steel by direct combustion, viz., "the method may be dismissed as quite out of the running, so far as steel is concerned. It requires the material to be in a state of impalpable powder—a condition impossible to most steels."

There is some truth in this quotation if "direct" combustion is taken to mean the ignition in a stream of air or oxygen only, but there is little or no worth in it in relation to the direct combustion of steel with suitable oxidising reagents.

The direct combustion of steel in the dry way is not only a practicable procedure; it is an eminently desirable one, where correct results must be rapidly obtained and the sample is either in such a physical state or contains such elements as invalidate Eggertz's colour test.

The estimation is made as follows:—Throw the borings on to a sieve having 20 meshes to the lineal inch, which is fitted into another having 40 meshes to the lineal inch. From the portion which passes through the one and not through the other, weigh out 2·5 grms. and mix with 6 grms. of red-lead (the blank of which on 10 grms. should not exceed 0·010 grm.) by shaking in a small weighing bottle. Transfer the mixture to a porcelain boat (4 × $\frac{3}{4}$), and place in the water-bath until ready for ignition.

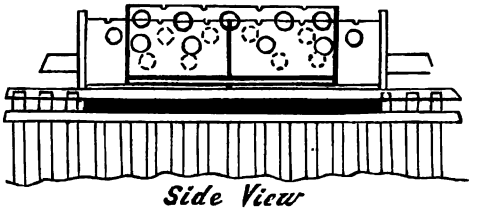
The combustion is conveniently made in a porcelain tube (20 × 1$\frac{1}{2}$), containing CuO for a few inches of its length and fitted at one end with means of purifying the incoming air or oxygen, and at the other end with a simple $CaCl_2$ drying tube. Lead chromate for absorbing sulphur compounds, or anhydrous $CuSO_4$ pumice for absorbing HCl, as in the ordinary combustion train, can be dispensed with, as the former never escape from the boat and the latter are not present. When the combustion-tube is at a red heat and the KHO bulb in its place, then insert the boat and quickly replace the stopper. There is no noteworthy evolution of gas due to a reaction between the sample and reagent such as takes place when ferrochromiums are suddenly heated in this way, and therefore, after having attached the aspirator and turned the taps full on, the combustion needs no further attention. With a good hot furnace combustion is complete in half-an-hour, even when air only (2$\frac{1}{2}$ litres) has been passed through. If the attached KHO bulb is so warm that it increases 4 or 5 milligrms. on standing in the balance case, it is best to work two bulbs alternately, but otherwise the bulb is re-weighed and replaced and a fresh combustion proceeded with. When drawn from the hot tube the contents of the boat are soft and can be easily scraped out with a suitably shaped piece of metal. The boat may then be used over and over again, but it should show no fine cracks or other signs of local weakness before being used, as it is then liable to break during ignition and mess the inside of the tube. Besides being otherwise objectionable, a coating of litharge increases the original tendency the tube has to crack on being suddenly heated.

For direct combustion a hot furnace is necessary. I use one of the Bunsen type, in which the customary toothed fire-clay tiles are replaced by asbestos arches. These enable a very high temperature to be obtained from only a modesate gas supply; particulars of their construction are given by Jervis (Assistant in this Laboratory) in the Chemical News, vol. lxxvii., p. 5. The figure shows a side view and section of the arrangement when a second larger cover is used to increase the heat over the portion of the tube occupied by the boat.

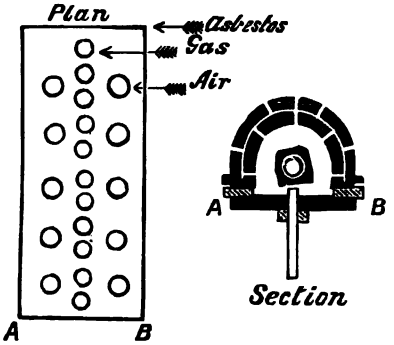
It may be asked if there is not some danger of the sievings from a sample containing more or less than its due proportion of carbon. I believe no source of error is to be feared with the medium sievings than attaches to the weighed amount taken in the ordinary way from the packet of coarse and fine. Special tests made on a series of standard steels showed that the carbon percentage with the coarse and fine portions were practically the same. The figures were:—

On coarse borings.	Through 30-mesh sieve.
0·136	0·120
0·457	0·457
0·758	0·754
1·060	1·036
1·243	1·243
2·286	2·291

Although sieving seems desirable, in order that material uniform in size and requiring about the same time to burn may be obtained, it must be said that any class of



Side View



borings made with ordinary care are completely decarbonised on heating with red-lead, but an extra fifteen minutes should be allowed in order to decompose any extra thick pieces. By still further increasing the period of ignition, chunks as large as peas and strips of sheet steel have been satisfactorily burned.

Below are given some results compared against those by dissolving in an acid solution of copper-ammonium chloride (5 per cent HCl), filtering, and ignition of the carbonaceous residue. (See Table, next column).

Direct combustion is particularly adapted to the estimation of carbon in special steels, such as tungsten and chromium-tungsten self-hards. As these borings are always thin, no sieving is ever necessary.

Attention has frequently been called to the fact that with ordinary carbon steels lower results are obtained with neutral solutions than with an acid solution of the double copper salt, and this is explained either by loss of hydrocarbons on dissolving or by the solubility of the residue in the menstruum. So far as steels containing special elements that are likely to exist as carbides are concerned, it seems equally important to point out that an acid solution of the double copper salt, as well as the acid (HCl) used subsequently for washing, may decompose or dis-

Acid solution of double copper salt.	Direct combustion.
Per cent C.	Per cent C.
0·15	0·15
0·19	0 19
0·23	0·25*
0·27	0·27
0·49	0·48
0·58	0·57
0·70	0·71
0·93	0·94
1·27	1·29
1·39	1·38
1·45	1·44
1·55	1·54
1·87	1·88

* Chunks as large as peas, specially picked out.

solve a portion of the metallic carbides. A friend using the direct combustion always finds this method to give the higher results, as much as 0·10 per cent.

The filtration of this class of steel, especially after forging, &c., on account of the fine state of division of the residue and its slimy nature, is somewhat troublesome, for as soon as the washings lose their acidity the residue comes through the filter. To overcome this difficulty, and at the same time the (probable) decomposing action of the hydrochloric wash, pure methylated spirit is used in this laboratory for washing, with extremely satisfactory results; although, on the average, the direct combustion gives the slightly higher—and probably more correct—figures.

Residue washed with methylated spirit.	Direct.
Per cent C.	Per cent C.
0·42	0·45
0·63	0·65
0·86	0·89
1·06	1·10
1·20	1·22
1·25	1·27
1·34	1·38
1·42	1·44
1·64	1·66
1·88	1·90
2·08	2·08
2·50	2·53

This direct estimation of carbon in steels was first tried in this laboratory in 1898. Since then at least two hundred of the results thus obtained have been checked by the ordinary process, and while, with the exception of special steels just named, the results have been in agreement, there were three or four samples of steels which were higher by the direct combustion without any assignable reason.

The Laboratory,
Messrs. Thomas Firth and Son, Lim.,
Sheffield.

Volumetric Estimation of Syrup of Iodide of Iron.—E. Rupp.—To effect this estimation, we add to 5 grms. of the syrup, diluted with water, acidulated with sulphuric acid, a solution of permanganate of potash at $\frac{1}{10}$th until the colour is persistent; after about two or three hours, potassic iodide is added; it is again left for an hour, and the iodine then titrated with one-tenth normal hyposulphite. We get, in fact:—

1. $10FeI_2+6MnO_4K+24SO_4H_2=$
$=20I+6SO_4Mn+5(SO_4)_3Fe_2+3SO_4K_2+24H_2O.$
2. $5(SO_4)_3Fe_2+10KI=10I+10SO_4Fe+5SO_4K_2.$

These equations show that $10FeI_2$ gives 30I; 1 c.c. of hyphosulphite corresponds to 0·2066 per cent of FeI_2 in the syrup.—*Arch. de Pharm.*, vol. ccxxxviii., p. 159. [It would be interesting to know what precautions the author takes to prevent or to allow for the reduction of the permanganate by the sugar].

ON THE DETECTION OF SMALL QUANTITIES OF ARSENIC IN FOODS, ESPECIALLY IN BEER.

By J. C. BERNTROP, Chemist at the Chemical Laboratory of the Municipal Sanitary Service of Amsterdam.

THE following simple method for detecting small quantities of arsenic in beer, &c., has been employed by me:—*One litre* of the beer is taken, a few drops of bromine added, and the mixture well shaken in order to convert the arsenious acid into arsenic acid. The liquid is allowed to stand for twenty-four hours, and then made strongly alkaline with ammonia. After adding 5 c.c. of a saturated solution of sodium phosphate, and 10 c.c. of the ordinary magnesia mixture, the liquid is allowed to stand for twenty-four hours in a moderately warm place. The precipitated ammonium-magnesium phosphate carries down with it the arsenic as ammonium-magnesium arsenate.

The clear liquid is poured off, and the precipitate transferred to a filter as completely as possible, then dissolved from the filter with 50—100 c.c. of warm dilute sulphuric acid (20 per cent).

The solution is run into the flask used for the precipitation, to dissolve the remainder of the crystalline precipitate adhering to the side of the flask.

The solution is heated in a Kjeldahl flask, small quantities of nitric acid being added from time to time until the liquid is totally decolorised and sulphuric acid begins to escape. This treatment is necessary in order to destroy the albumenoids which otherwise would occasion the development of hydrogen sulphide when using the Marsh or the Gutzeit test, for which the liquid is now ready.

The method has proved to be likewise useful in the examination of other drinks and of food.

With great interest all chemists will have read the "Report of the Joint Committee of the Society of Chemical Industry and of the Society of Public Analysts on the Detection and Approximate Estimation of Minute Quantities of Arsenic in Beer, Brewing Materials, Foodstuffs, and Fuels."

I think that as my method allows the use in one manipulation of *one litre* of beer—the committee experiments on *ten or twenty c.c.* of beer—it may be a useful addition to theirs.

Dionine.—This substance, prepared by E. Merck, of Darmstadt, is described as a mild and pleasant substitute for morphia and codeia; it is a derivation of morphia, having the composition expressed by the formula $C_{19}H_{23}NO_3HCl+H_2O$. It appears as a white, odourless, micro-crystalline powder of bitter taste, melting at 123—125°, and very easily soluble in water. The aqueous solutions possess a neutral reaction, and keep for a long time, clear and free from decomposition. Owing to its neutral character injections of dionine are not painful, whilst its greater solubility enables it to diffuse more readily into the circulation, and develope its action more quickly than any morphia derivative yet employed, it is also devoid of any toxic properties, and, according to the unanimous opinion of all observers, the taking of dionine never becomes a habit. According to experimental investigations by Hoff, intravenous injections of dionine cause immediate retardation of the respiration, and longer expirations and inspirations; under its influence the atmospheric air remains longer in contact with the capillary vessels of the lungs; the oxidation of the blood and the removal of the carbonic acid is therefore more complete, and the effect of the ventilation of the lungs increased.

Crystallisation of Peroxide of Iron.—Alfred Ditte.—It is known that when a mixture of hydrated iron sulphate and common salt is calcined, the oxide which results from the destruction of the crystalline ferrous salt in the middle of the fused salt can be separated out by repeated extraction of the fused mass with hot water. The author discusses the mechanism of this reaction.—*C. R.*, cxxxiv., No. 9.

ON THE ANALYSIS OF PYRITIC RESIDUES.

By A. MINOZZI.

At the present time the sulphuric acid industry uses, almost entirely, sulphur from pyrites, a mineral composed essentially of bisulphide of iron. Pure pyrites contains 53·3 per cent of sulphur and 46·7 per cent of iron, but the natural product is rarely pure. It always contains gangue, copper, zinc, lead, arsenic, selenium, antimony, peroxide and protoxide of iron, and sometimes manganese, titanium, &c. Finally, phosphorus is often met with. The operation of roasting for the production of sulphurous anhydride leaves a residue (called *Purpurerz* or *Kiesabbrände* in Germany, and *purple ore* in England), in which the iron exists almost entirely in the form of the sesquioxide.

If the pyrites contains reasonable proportions of zinc or copper the residue is utilised for the extraction of these metals; if this is not the case, it forms, at all events for the Italian manufacturer, a most embarrassing by-product, on account of the large cost of transport. The idea of utilising these residues is by no means new. Metallic iron is present to the extent of 56 to 65 per cent, and as they only, as a rule, contain a small amount of phosphorus (less than 10 grms. per 100 kilogrms.) these residues form an excellent iron ore, and even yield a pig-iron of good quality.

I have recently had occasion to analyse various samples of pyritic residues with the idea of using them as iron ores. One sample contained 0·92 per cent of copper, another 0·85 per cent of titanium, and all contained 6·32—8·47 per cent of silica, 0·85—2·16 per cent of sulphur, 0·16—0·37 per cent of lead, and 0·005—0·021 per cent of phosphorus.

The proportion of iron varied from 57·12 to 60·81 per cent. With the exception of one only, which only contained traces, all the samples examined were free from arsenic. The estimation of the alkaline earthy metals was not made.

Every analyst knows how difficult it is to dissolve pyritic residues. Lunge recommends, for estimating the sulphur by his method, pulverising the material in an agate mortar. Borntraeger (*Zeit. Anal. Chem.*, xxxviii., 774) having observed that aqua regia does not dissolve calcined ferric oxide easily, but that this body is easily dissolved by hydrochloric acid containing free chlorine, iodine, or bromine, or peroxide of hydrogen or nascent hydrogen, advises the addition of peroxide of manganese free from iron to the hydrochloric acid. Under these conditions the chlorine which is evolved rapidly dissolves the ferric oxide. I have attempted to dissolve pyritic residues in hydrochloric acid containing chlorate of potassium, but even after twenty-four hours I have never obtained complete solution. It follows from the above that the ordinary wet methods of analysis must be abandoned if we wish for satisfactory results, and I hope that the methods I am about to describe for obtaining exact results as rapidly as possible will prove to be of use.

Sulphur.—For the estimation of sulphur I follow a modification of the fusion method described by J. Deutecom (*Ibid.*, xix., 313) and applied by the author to pyrites. I had, in fact, observed that this method is superior to and more rapid than fusion with a mixture of carbonate of soda and nitrate of potassium. Two grms. of the pyritic residue are weighed out accurately in a platinum crucible, and intimately mixed with 5 grms. of a mixture composed of 2 parts of anhydrous carbonate of sodium and 1 part of chlorate of potassium; the whole is then covered with 2 grms. of the same mixture. Although coal-gas of good quality is almost free from sulphuretted hydrogen, it is not safe to heat the crucible on a triangle; I prefer a sheet of asbestos card with a circular hole in the middle, which will just hold the covered crucible. By this arrangement the products of combustion from the source of heat do not come into contact with the fused mass, and therefore cannot falsify the results. We begin by heating gently until the top layer of pure mixture is fused; this should take about half-an-hour. The heat is then gradually increased, and after another ten minutes the gas is turned full on; the mass, from being pasty, becomes solid, then reverts to a semi-fluid condition, and then becomes pasty again. Care must be taken to reach this point so as to be certain of the complete decomposition of the perchlorate. After cooling, the crucible with its contents and the cover are thrown into a beaker containing about 300 c.c. of warm water, and allowed to digest, warm, until the whole of the melted mass is disaggregated. The clear liquid is then decanted through a small filter and collected in an Erlenmeyer matras with a flat bottom, of about 1 litre capacity. The residue is washed by decantation, five or six times, with a 2 per cent solution of dry carbonate of sodium, using about 50 c.c. of the solution for each washing; it is then finally heated to boiling. The last wash-waters should no longer give any sulphuric acid reaction, and only traces of hydrochloric. The wash-waters added to the principal liquor make about 600 c.c. Forty c.c. of 10 per cent hydrochloric acid are added, and the whole heated to boiling to completely remove all the carbonic acid, and the sulphuric acid is precipitated by 10 c.c. of 10 per cent chloride of barium, taking the usual precautions.

Silica, Lead, Titanium, Copper, and Iron.—Five grms. of the pyritic residues are placed in a platinum crucible of about 30 c.c. capacity, and while heating gently about 25 grms. of bisulphate of potassium are gradually added. The temperature is gradually raised until the melted mass is homogeneous. After cooling, it is treated with 500 c.c. of 2 per cent sulphuric acid and filtered, the filtrate being collected in a graduated litre flask. The residue is washed with boiling water, slightly acidulated with sulphuric acid, then dried, calcined, and weighed. It is then moistened with 5 c.c. of 10 per cent sulphuric acid and 20 per cent hydrofluoric acid, and carefully evaporated. The last traces of sulphuric acid are neutralised by the addition of a small quantity of carbonate of ammonium, after which it is calcined strongly until the weight remains constant. The difference between the two weights represents the amount of silica present in the original sample.

The residue, which still contains a little ferric oxide, is again fused with bisulphate of potassium, dissolved in sulphuric acid, and added to the principal solution, which is then made up to a litre. The insoluble part consists mainly of sulphate of lead; it is treated several times with a concentrated boiling solution of acetate or tartrate of ammonium; the lead is precipitated by sulphuretted hydrogen, and estimated in the form of sulphate after having been re-dissolved in nitric acid.

As for the principal solution, 100 c.c. are taken for the estimation of the iron, and the remainder, which serves for the estimation of the other elements, is evaporated down to about 300 c.c. To estimate the titanium in iron ores, Brakes (*Journ. Soc. Chem. Ind.*, xviii., 1097) reduces the hydrochloric solution of the ore with a solution of sulphurous acid; Baskerville (*Moniteur Scientifique*, Oct., 1900, p. 697) treats the hydrochloric solution with a current of gaseous sulphurous anhydride, but I prefer a solution of sodium bisulphite, prepared by means of a 50 per cent solution of crystallised carbonate of soda. In this manner I obtain a solution containing 21 per cent of available sulphurous anhydride.

The concentrated portion of the principal liquor is placed in an Erlenmeyer matras of about 600 c.c. capacity, and neutralised with ammonia; it is then made slightly acid with hydrochloric acid, and treated with bisulphite of sodium; the titanic and phosphoric acids are precipitated in a slightly coloured flocculent form. This precipitate is collected on a filter, washed with boiling water, and then fused with about 2 grms. of a mixture composed of 2 parts of carbonate of sodium and 1 part of nitrate of potassium. The melted mass is taken up with 200 c.c. of boiling water, the phosphate of sodium formed is dis-

solved, while the residue of titanate of sodium and ferric oxide is collected on a filter. This is fused with bisulphate of potassium, and the treatment with bisulphite of sodium is repeated; in this manner perfectly pure, white titanic acid is obtained, which is calcined and weighed. As for the phosphoric acid remaining in solution, it is precipitated in the form of phospho-molybdate of ammonium, and estimated in the form of pyrophosphate of magnesium.

The solution, thus freed from titanium and phosphorus, is treated with a little bisulphite of sodium, acidulated with 50 c.c. of 10 per cent hydrochloric acid, and submitted while warm to the action of a current of sulphuretted hydrogen.

The precipitated sulphides, after washing with sulphuretted hydrogen water, are calcined in a small porcelain crucible, and the residue is dissolved in nitric acid. The solution obtained is made alkaline by ammonia, then treated with a small quantity of carbonate of ammonium, and heated on the water-bath for some hours.

It is then filtered, and the copper is estimated in the filtrate either by the electrolytic method, or gravimetrically, by precipitating and weighing it in the form of cuprous sulphide.

In 100 c.c. of the principal sulphuric solution the iron is estimated by permanganate, either directly or after separation in the state of basic carbonate if the pyritic residues contain manganese, zinc, and other metals.—*La Chimica Industriale*, vol. ii., p. 248.

ESTIMATION OF THE PHOSPHORIC ACID IN PHOSPHATES.

By J. A. MULLER.

The estimation of the titration value of the uranium solution serving for the volumetric estimation of the phosphoric acid in phosphates is often done by means of a titrated solution of crystallised phosphate of sodium. The facility with which this salt loses moisture does not, however, admit of any great accuracy. The crystallised salt of phosphorus, $PO_4HNaNH_4.4H_2O$, is much more stable in this respect. This salt, pulverised and spread in a thin layer in a crucible, only loses 0·15 per cent of its own weight in the space of four hours at 18° in the air, of which the hygrometric degree was equal to 0·5, while, under the same conditions, the crystallised phosphate of sodium loses 2·40 per cent. Even when placed over phosphoric anhydride, this crystallised salt of phosphorus only undergoes a slight loss of weight, 1 68 per cent after twenty-four hours at the ordinary temperature, while crystallised phosphate of soda ends, in this case, by becoming almost, if not quite, dehydrated.

However, this salt of phosphoric is far from possessing the stability of crystallised dicalcic phosphate, $(PO_4)_2Ca_2H_2.4H_2O$, which, when freely exposed to the air and under the preceding conditions, or even over phosphoric anhydride, does not vary in weight at all at the ordinary temperature. We can also recommend a nitric solution of this phosphate—in which the proportion of phosphoric acid has previously been determined—to determine the value of the uranium solution serving for the titration of commercial phosphates.

To prepare the crystallised dicalcic phosphate, we gradually add a cold dilute solution of disodic phosphate to a dilute solution of pure chloride of calcium until the almost complete precipitation of this metal is effected. The precipitate, which is gelatinous at the moment of its formation, rapidly becomes crystalline; it is easy to wash it completely. When washed, it is thrown on flat plates and dried in the oven at a temperature of about 70°. Theoretically, the salt thus obtained should contain 41·27 per cent of P_2O_5. The estimation of this substance in the state of magnesic pyrophosphate, after precipitation in the form of phosphomolybdate and ammonio-magnesian phosphate, gave 41·45 and 41·38 per cent of P_2O_5. By estimating the phosphoric acid by means of citro-magnesian solution, I found 41·55 and 41·29 per cent.

With regard to this latter method of estimation, an important remark may be made. The first precipitate of ammonio-magnesian phosphate—obtained by adding the citro-magnesian solution, then an excess of ammonia to the nitric solution of the phosphate—contains a small quantity of calcium. To separate this, the precipitate is re-dissolved—after separation from its mother-liquor—in nitric acid, a small quantity of citro-magnesian solution, chlorhydrate of ammonium, and then ammonia are added; after twelve hours, it is filtered, and the precipitate washed with ammoniacal water, &c. It is advisable to conduct this last operation in a platinum crucible, which will serve to calcine the ammonio-magnesian phosphate.

It may also be remarked that the precipitate obtained by adding the magnesian mixture to an ammoniacal solution of phosphomolybdate contains, or may contain, a little molybdenum, which can be separated by re-dissolving the precipitate in hydrochloric acid and re-precipitating by ammonia in the presence of a little of the magnesian mixture.—*Bull. Soc. Chim.*, Series 3, vol. xxv., No. 23.

THE ATOMIC WEIGHT OF ANTIMONY.*

By G. CLAUSEN FRIEND and EDGAR F. SMITH.

Knowing that antimony oxide could be completely expelled from its combinations in a current of hydrochloric acid gas, it appeared probable that a new ratio might be established for antimony, by exposing potassium antimonyl tartrate to the action of this gas. It will be recalled that by this procedure the atomic weights of molybdenum and arsenic had been previously determined in this laboratory. In these particular instances sodium molybdate and sodium pyroarsenate were exposed in porcelain boats, at a moderate heat, to the action of the gas, and from the weight of the residual sodium chloride the respective atomic weights were calculated. The method of work adopted with these metals was pursued with potassium antimonyl tartrate, but it was soon discovered that as carbon dioxide and water escaped the salt swelled up and was projected from the boat, so that a double crucible was substituted for the latter. On trial this device proved to be perfectly satisfactory, and it was possible in a crucible No. 00, from 1 to 1½ inches in height, to operate with as much as 3 grms. of material. The appended sketch represents the apparatus in detail.

A steady stream of hydrochloric acid gas was generated in the large flask by acting with pure, concentrated, sulphuric acid upon hydrochloric acid of like character. The gas was sufficiently dried by its passage through sulphuric acid and a column of calcium chloride, and then introduced through a porcelain delivery tube into the crucible. The smaller crucible was supported in the larger one upon a perforated porcelain plate. The lid of the larger crucible had two openings, one for the entrance, and the other (A) for the exit of the excess of acid gas. Platinum crucibles should not be used; they are very severely attacked by the acid vapours.

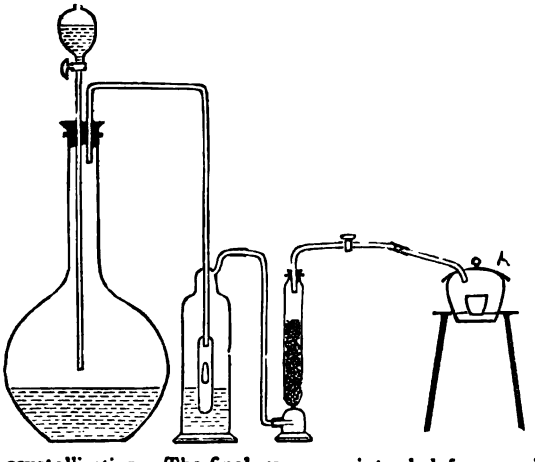
In the experiments the dry gas was passed over the salt in the smaller crucible for half-an-hour, before any heat was applied. At the expiration of this period a small flame was placed under the crucible, and it was heated to 150° for two hours, after which the temperature was gradually increased until the outer crucible showed a dull red colour. This temperature was maintained until all of the volatile matter was expelled, when a stream of oxygen was substituted, for half-an-hour, for the acid. Most of the carbonaceous material was removed in this

* Contribution from the John Harrison Laboratory of Chemistry. From the *Journal of the American Chemical Society*, xxiii., No. 7.

way, when the acid vapour was again introduced, and the crucible contents allowed to cool in it. As the residual potassium chloride contained some carbon, it was dissolved in water, and the solution filtered. The filtrate, with washings, was collected in a weighed platinum dish and evaporated upon a water-bath. The dish was supported upon a perforated glass plate. The dry potassium chloride was finally heated in an air-bath to 150°, then removed and gently heated over a flame to expel the last traces of moisture. On cooling, the salt was weighed. Two experiments made with potassium antimonyl tartrate, not especially purified, and with an ordinary balance and weights, gave the following results:—

	Salt.	Potassium chloride.	Atomic weight of antimony.
	Grms.	Grm.	
1	2·0358	0·4691	120·44
2	2·5919	0·5973	120·41

This concordance in result and the ease with which the method could be executed, led us to prepare and purify large quantities of potassium antimonyl tartrate. To this end the purest commercial salt was re-crystallised ten times, the first fraction only being used in each subsequent



crystallisation. The final 50 grms., intended for experiment, were dried at 150° for a period of sixteen hours. A weight was taken, and the mass then again heated, cooled, and re-weighed. There was no variation in the weight. This material was carefully tested for impurities (*e.g.*, arsenic, sodium, silica, &c.), and was found free from them. Portions of it were then acted upon as outlined in the preceding paragraphs.

TABLE A.

	Potassium antimonyl tartrate.	Potassium chloride.	Atomic weight of antimony.
	Grms.	Grm.	
1 ..	1·19481	0·27539	120·345
2 ..	1·57004	0·36186	120·359
3 ..	2·00912	0·46307	120·351
4 ..	2·04253	0·47073	120·379
5 ..	2·16646	0·49935	120·341
6 ..	2·25558	0·51982	120·385
7 ..	2·61255	0·60215	120·350
8 ..	2·95272	0·68064	120·311
		Mean =	120·353

Maximum = 120·385
Minimum = 120·311

Difference 0·074

The balance used in the subsequent experiments was constructed for atomic weight work by Troemner. It is sensitive to $\frac{1}{20}$ of a milligrm. with or without load. The weights of brass and platinum were carefully calibrated. All weighings were reduced to the vacuum standard. The specific gravity of potassium chloride was taken as 1·995 and that of the tartar emetic as 2·6 (Beilstein). The atomic weights used in the calculations were O=16, H=1·008, C=12, K=39·11, and Cl=35·45. The results obtained with the pure material and with the observance of all the necessary precautions are shown in Table A.

The barium and silver antimonyl tartrates crystallise well, and the hope was entertained that these salts might also be included in the circle of experimentation, but thus far the results with them have not been satisfactory.

ON PYRITE AND MARCASITE.*

By H. N. STOKES.

(Continued from p. 115).

X. Miscellaneous Specimens and Specimens of Doubtful Nature.

With the aid of the pyrite-marcasite curve we are now in a position to determine the quantitative composition of specimens of pyrite and marcasite in regard to which there is some uncertainty. The supposed nature of the specimens, according to the geologist, mineralogist, or dealer from whom they were obtained, is stated, as well as the composition established by my method. It is to be understood that the figures express the relative amount of pyrite and marcasite on a scale of 100, impurities being excluded. In this connection I wish to acknowledge the generosity of Dr. A. A. Julien, who has placed at my disposal a number of the identical specimens described by him in his valuable paper repeatedly referred to above. As the study of these specimens is especially important in confirming or refuting his hypothesis (see Section VI.), I have grouped them together at the end of this section, and have in each case quoted his original description and specific gravity determination, comparing in parallel columns the results obtained by the specific gravity and the oxidation methods.

Notes to Table A.

No. 15. Nearly spherical concretion from coal; locality unknown. Weight, 9·2 grms. Very hard. The structure is coarsely fibrous and radial, with concentric shells of pyrite and marcasite, easily distinguishable by their colour.

No. 16. Pyritised ammonite from Folkestone, England. The walls of the cavities are encrusted with pyrite crystals, but the nature of the mass cannot be determined from the colour.

No. 17. Concretion from Red Cloud Mine, San Juan County, Colorado, supposed to be marcasite. It consists of a finely fibrous incrustation on rhyolite, varying in thickness from 1 to 10 m.m., with mammillary surface, and with a banded structure and cleavage perpendicular to the fibres. The banded structure is due to the enclosure of quartz and a black substance which, judging from the presence of a little copper, lead, and arsenic, may be galena, arsenopyrite, and chalcocite. The latter would account for the high oxidation coefficient, which could not be caused by the first two (see p. 92). Carefully selected material gave a specific gravity of 4·563.

No. 18. The occurrence of this material is thus described by Mr. Lindgren, by whom it was submitted for a determination of the yellowish grey material:—"This specimen is derived from the Present Need Mine, Quartzburg district, Grant County,

* From the *Bulletin of the U.S. Geological Survey*, No. 186.

TABLE A.—*Quantitative Composition of Specimens of Doubtful Nature.*

(Figures in parenthesis were determined by the indirect method).

No.	Original designation, &c.	Locality.	p.	Pyrite. Per cent.	Marcasite. Per cent.
15.	Concretion from coal	Unknown	30·1	63·0	37·0
16.	Pyritised ammonite..	Folkestone, England	46·9	88·0	12·0
17.	Fibrous marcasite	Red Cloud Mine, Colorado	65·7	100·0	—
18.	Pyrite with marcasite	Quartzburg district, Oregon ..	38·6	78·0	22·0
19.	Marcasite	Chautauqua Tunnel, Idaho.. ..	51·3	93·5	6·5
20.	Marcasite	Garfield Tunnel, Idaho	19·8	30·5 (34·4)	69·5 (65·6)
21.	Marcasite nodules	South Dakota..	60·3	100·0	—
22.	Marcasite (selected)..	Littmitz, Bohemia	19·5	29·5 (27·8)	70·5 (72·2)
23.	Marcasite (selected)..	Crow Branch Mine, Wisconsin ..	18·6	26·0 (31·0)	74·0 (79·0)
24.	Fibrous marcasite	Sunshine, Colo.	52·2	(*a*) 94·0	(*b*) 6·0
25.	Marcasite after pyrite	Folkestone, England	55·4	97·0	3·0
26.	Pyrite ore	Rio Tinto, Spain	65·7	100·0	—
27.	Pyrite octahedra	Unknown	80·4	100·0	(*c*)

(*a*) Or more. (*b*) Or less. (*c*) Much cobalt.

TABLE B.—*Quantitative Composition of Julien's Specimens.*

(The numbers in parentheses are those given in Julien's paper).

	Number.	Locality.	Density.	p.	Composition deduced from p.	Composition according to Julien, deduced from density.
28.	(Pyrite 1).. ..	Galena, Ill...	5·015	60·3	Pyrite.. .. 100·0	Much Marcasite.
29.	(Pyrite 96) ..	Franklin, N.J.	4·856	59·0	Pyrite.. .. 99·5	Pyrite.. .. 27·51 Marcasite .. 72·49
30.	(Pyrite 98) ..	Somerville, Mass. ..	4·843	59·6	Pyrite.. .. 99·5	Pyrite.. .. 21·18 Marcasite .. 78·82
31.	(Pyrite 101) ..	Monroe, Conn.	4·819	60·7	Pyrite.. .. 100·0	Pyrite.. .. 9·38 Marcasite .. 90·62
32.	(Pyrite 106) ..	Bastrop, Texas	4·791	67·7	Pyrite.. .. (*a*) 100·0	Pyrite.. .. 1·00 Marcasite .. 99·00
33.	(Marcasite 1) ..	Cumberland, England.	4·987	42·9	Pyrite.. .. 83·0 Marcasite .. 17·0	Pyrite.. .. 89·45 Marcasite .. 10·55

(*a*) Contains nickel.

Oregon. The vein, carrying chiefly gold, partly in free form, partly associated with sulphides, is contained in diabase and diabase-porphyry. The ore minerals are normal yellow pyrite, a soft yellowish grey material supposed to be marcasite, zinc blende, and chalcopyrite." The iron sulphides are easily separated from the rock and other sulphides by hydrofluoric acid. The yellowish grey material, which is closely veined with pyrite, could not be separated from the latter, but the oxidation coefficient of the mixture indicates that it is marcasite. The portion examined contains a trace of copper and lead and no zinc.

No. 19. This is thus described by Mr. Lindgren :—"This specimen is taken from the dump of the Chautauqua Tunnel, at De Lamar, Owyhee County, Idaho. The specimen consists of quartz, upon which there is a crust of pyrite. The quartz itself contains arborescent forms of a mineral supposed to be marcasite, and described as such in *Twentieth Annual Report, U.S. Geological Survey*, Part III., p. 130." The mineral in question was easily separated from the quartz and the mass of the pyrite by digestion with hydrofluoric acid. It formed dark coloured tabular masses, presenting no definite crystalline form, and containing traces of copper and lead with a notable amount of arsenic. The oxidation coefficient (51·3) shows that it consists of at least 93·5 per cent of pyrite, but the proportion thus determined is somewhat too low, owing to the influence of the arsenic.

No. 20. Thus described by Mr. Lindgren :—"This specimen is taken from the Garfield Tunnel, 250 feet from the portal, near De Lamar, Owyhee County, Idaho, and is described in *Twentieth Annual Report, U.S. Geological Survey*, Part III., pp. 131 and 171. The specimen consists of a soft white material, which, according to analysis, is probably kaolinite mixed with sericite. It contains arborescent forms of marcasite." The supposed marcasite was isolated by hydrofluoric acid. It could not be definitely described as such from the colour and crystalline form. As the oxidation coefficient (19·8) was too high for pure marcasite, a portion was mixed with one-ninth its weight of pyrite, and then gave—

$$p = 22\,6 = 41 \text{ per cent pyrite.}$$

We have, then—

$$0{\cdot}9\,x + 10 = 41, \text{ or } x = 34{\cdot}4.$$

This corresponds to $p = 20{\cdot}6$, a number agreeing within permissible limits with the original determination. We have, therefore,—

Pyrite directly determined .. 30·5 per cent
Pyrite indirectly determined . 34·4 ,,

and the original conclusion of Mr. Lindgren is only in part confirmed. A little arsenic is also present.

No. 21. Material supposed to be marcasite, from South Dakota. Submitted by Dr. G. P. Merrill. The sample consists of spherules not over ½ m.m. in diameter, and so brittle as easily to be crushed by the finger. They had undergone vitriolisation to such an extent that the original substance was entirely concealed in the mass of sulphate. The oxidation coefficient shows that the material is pyrite, and the analysis that some copper is present. The normal value of p indicates that the copper is probably in the form of chalcopyrite.

No. 22. Marcasite from Littmitz, Bohemia, consisting of flat deeply striated twins of marcasite on a nucleus which on fracture shows both marcasite and pyrite. Only the portions free from visible ad-

mixture of pyrite were taken for examination, and gave $\rho = 19{\cdot}5$. Admixture with one-ninth pyrite gave $\rho = 21{\cdot}0 = 35$ per cent pyrite. We have, then,—

Pyrite directly determined .. 29·5 per cent
Pyrite indirectly determined . 27·8 „

(Regarding the Littmitz marcasite, see Sadebeck, *Monatsber. K. Preuss. Akad. Wiss. Berlin*, 1878, vol. xx., p. 19; and Hintze, "Handbuch der Mineralogie," vol. i., pp. 732, 825).

No. 23. A specimen from Crow Branch Mine, Wisconsin, consisting of a compact nucleus of tin-white marcasite covered with crystals which are completely enclosed in elongated tubes of pyrite, the line of demarcation being clearly discernible on the fracture by the difference of colour. Selected portions free from visible admixture of pyrite were used, giving a specific gravity of 4·891. To determine the contents of pyrite the indirect method was used.

Pyrite directly determined .. 26·0 per cent
Pyrite indirectly determined . 31·0 „

As only the powdered material was available for the indirect determination, the difference is not surprising (see p. 82).

No. 24. Material from Sunshine, Colorado. Finely fibrous radial concretions in quartz, which on the freshly cleaned fracture show a faint banded structure. Supposed to be marcasite. A little lead and copper and much arsenic and antimony are present. As arsenic, and probably antimony, would lower the oxidation coefficient, the figure given for pyrite is too low, and the presence of marcasite is doubtful.

No. 25. A large flattened concretion from Folkestone, England, described as "marcasite after pyrite." The fracture is columnar and the surface is covered with rounded projections, apparently tetragonal pyramids, possibly of the type described by Penfield (*Am. Journ. Science*, 1889, Series 3, vol. xxxvii., p. 209). The colour on the freshly-broken surface exactly matches pyrite. I have received several other supposed marcasite concretions from Folkestone, all of which are clearly pyrite.

No. 26. Pyrite ore from Rio Tinto, Spain. The fragments are greyish, evolve some hydrogen sulphide with acids, and with the bromine hydrogen sulphide test (p. 115) show very finely disseminated copper sulphide.

No. 27. Brilliant untarnished octahedra in calcite, containing a large amount of cobalt and a little copper, which explains the abnormal oxidation coefficient.

Conclusions.

Of the above thirteen specimens five have been described as marcasite, though consisting nearly or entirely of pyrite, while three other marcasites are shown to contain a large amount of pyrite, which could not be detected without the aid of the oxidation method. As these samples were selected and examined without preconception as to their nature, it seems probable that a very considerable proportion of the concretions which exist in collections or are sold as marcasite are in reality pyrite, while not a few others, even when fairly well characterised by rhombic crystallisation, may contain inclosures of pyrite.

Dr. Julien's Specimens.

The following descriptions are in part quoted from Julien's paper:—

Notes to Table B.

No. 28. "Concretionary nodule. Marsden's diggings, Galena, Ill. No. 1 (fibrous core). Marcasitic pyrite. Very finely fibrous, pale brass-yellow and splendent." Analysis (Julien), SiO_2, 0·110; Pb, 0·188; As, 0·056. The fracture varies from fibrous to prismatic, the prisms being evidently elongated cubes, with terminations consisting of cube modified by pyritohedron and octahedron.

No 29. "No. 96. Marcasitic pyrite. Franklin, N.J. Sharp, brilliant, brass-yellow, striated pyritohedra, yellowish-white, and splendent on fracture." No admixture of marcasite crystals. The value of ρ shows that they are practically pure pyrite, the low density being probably due to inclosures.

No. 30. "No. 98. Marcasitic pyrite. Somerville, Mass. Very sharply defined, glittering yellowish cubes, sometimes distorted or rectangular, rarely striated, occasionally with octahedral planes upon their solid angles, yellowish white, and brilliant on fracture; scattered through a grey argillaceous slate."

No. 31. "No. 101. Pyrite. Monroe, Conn. Deeply striated, distorted, brilliant cubes of very pale brass-yellow colour, with modifications by the pyritohedron; yellowish white, finely granular, and splendent on fracture, and sometimes inclosing grains of white quartz."

No. 32. "No. 106. Marcasitic pyrite. Bastrop, Bastrop County, Tex. Dull and pale brass-yellow octahedra, rarely bright, very pale yellowish white, and brilliant on fracture; upon black granular crystalline hematite." The specimen contains a trace of arsenic, a strong trace of copper, and some nickel. It is the latter, possibly in conjunction with the copper, which causes the high oxidation coefficient.

No. 33. "No. 1. Marcasite. Cumberland, England. Hollow incrustation pseudomorphs after barite, implanted on a group of barite crystals. The crusts are greyish white to tin-white, and splendent on fracture, with surfaces drusy with pseudo-octahedrons or rhombic pyramids, whose smooth, rectangular terminal faces (the basal pinacoid OP) project but slightly; a few minute rhombic prisms also occur, and some hexagonal though apparently triangular twins." The supposed rhombic pyramids with basis are evidently combinations of cube and octahedron belonging to the 83 per cent of pyrite which it contains.

Discussion of Julien's Hypothesis.

The above list includes some of the most pronounced cases of "marcasitic pyrite" described by Julien—that is, material with the crystalline form of pyrite, but supposed to consist very largely of marcasite. The four specimens, Nos. 29, 30, 31, and 32, consist of characteristic pyrite crystals, and if the oxidation method be admissible they are, in fact, free from marcasite, yet they all show abnormally low densities. The only conclusion which can be drawn from this is that the density alone does not afford a reliable means of detecting the presence of marcasite in pyrite unless the absence of all other contaminating substances be first conclusively proved, and that isometric crystallisation in itself affords a proof, almost amounting to certainty, that the sulphide is actually pyrite. It is undoubtedly true that perfect pyrite crystals may enclose marcasite, a good case of which is the Crow Branch specimen, No. 23, but such cases are comparatively rare, and the inclosure can usually be detected by breaking the specimen, and carefully examining the colour. In case of doubt a determination of the density is practically of no value, and resort must be had to the oxidation method. I have examined some of the marcasite specimens from the same collection, as well as others which are supposed to contain pyrite, and in general the latter can be detected by its colour after careful cleaning with acid. I have shown that certain specimens of marcasite do contain pyrite enclosed in such small masses as not to be capable of detection in this way

(Nos. 22 and 23). Small, warty excrescences on marcasite crystals are frequently pyrite, and when the conditions controlling the formation of the two sulphides are practically in equilibrium, these are likely not only to form but to be completely overgrown and enclosed in the crystal of marcasite. A marcasite which occurs mixed with visible masses of pyrite is therefore likely also to contain such inclosures which cannot be seen and whose presence can be detected only by the indirect oxidation method. That both pyrite and marcasite with abnormal density are essentially prone to decomposition is doubtless true, and while in certain cases this may be traced to an admixture of the two, in others it is due to inclosures of another nature. The lack of homogeneity not only permits the existence of fissures admitting air and moisture, but the formation of these is in such cases promoted by unequal expansion and contraction.

Fibrous marcasite is so often referred to that it may well exist, but so far as my somewhat limited range of examinations goes, a fibrous structure affords absolutely no presumption in favour of a specimen being marcasite; on the contrary, fibrous pyrite appears to be much more common than fibrous marcasite, the latter in concretions tending rather to assume the coarsely columnar structure.

XI. Paramorphism.

The literature of the paramorphism of pyrite and marcasite is exceedingly scanty, and such as there is lends no support to the view that true transformation paramorphs exist. Those described are apparently either replacement or incrustation pseudomorphs, the true nature of which is shown by careful inspection. Wöhler (*Liebig's Annalen*, vol. xc., 256) attempted, without success, to convert marcasite into pyrite and pyrite into marcasite by heating for four hours at about 400°—experiments which are well worth repeating with an extension of the time and with the aid of the oxidation method. Julien's assumption of extensive paramorphism is scarcely longer tenable in view of the results described above, although the enclosure of considerable portions of pyrite in some specimens of marcasite lends some support to it. While theoretically possible, the attractive hypothesis that marcasite gradually changes to pyrite without re-solution, and that the porosity of certain pyrites can be thus explained, is thus far without a sound basis, either of experiment or of observation. It is intended to carry out experiments on this point in the immediate future.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, February 19th, 1902.

Prof. J. Emerson Reynolds, Sc.D., V.P.R.S., President, in the Chair.

Messrs. E. A. Lewis, R. A. Berry, H. W. Crowther, and H. O. Jones were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Cyril Bergtheil, Checkley, Red Hill, Surrey; Ernest Boardman, 41, Knowsley Road, Smithills, Bolton; Charles Benson Davis, 218, West 134th Street, New York, U.S.A.; Arthur Hopwood, 49, Stanley Street, Tunstall; Jonathan Hugh Roberts, Oban Gwys, Swansea; Stephen Herbert Trimen, 61, St. John's Park, N.; William Wright Tunnicliffe, Newhall, Burton-on-Trent; William Charles Wain, Mercantile Explosive Dept., Sydney, N.S.W.

It was announced that the following changes in the Officers and Council were proposed by the Council:—

As Vice-Presidents—Prof. Meldola, F.R.S., and Prof. Frankland, FR.S., *vice* Mr. Groves, F.R.S., and Prof. Purdie, F.R.S.

As Foreign Secretary—Prof. W. Ramsay, F.R.S., *vice* Prof. Meldola, F.R.S.

As Ordinary Members of Council—Mr. J. E. Marsh, Dr. J. A. Voelcker, Dr. A. Harden, and Dr. Lewkowitsch, *vice* Mr. Fenton, F.R.S., Mr. Howard, Mr. Pope, and Dr. Crossley.

Mr. A. C. Chapman, Dr. Hewitt, and Dr. Thorne were appointed to audit the Society's accounts.

A ballot for the election of Fellows was held, and the following were subsequently declared duly elected:—Keith Benham Benham; Walter Geoffrey Black; Harry Burrows, Ph.D.; T. K. Buxy, M.A.; Frederick E. Catchpole, B.Sc.; Matthew Bradbury Challen; Kenneth Macomb Chance, B.A.; William Clifford; James Ward Daniels; Frederick Davis, B.Sc.; Evelyn Andros De la Rue, B.A.; William Dennis; John Kemp Smith Dixon; Charles Dorée, M.A.; Lewis Eynon; Alfred Vincent Elsden; Eugéne Arthur Fasnacht; Frederick Ferrand; John Oliver Ferrier; Clarence J. Green, B.Sc.; Paul Haas, B.Sc., Ph.D.; Noël Heaton, B.Sc.; Eugene Edwin Hennesey, B.A., B.Sc.; George Frederick Holdcroft; W. Holdsworth Hurtly, D.Sc.; William Brannan Jackson; David S. Smith Jardin; Selwyn Philip James Lavelle; Harry Lucas; Ernest Bowman Ludlam, B.Sc.; John Ross Mackenzie; William Maitland, B.Sc.; Francis Martin; Francis Hylton Molesworth; Alfred Holley Mundey; Allan Ogilvie; Sydney Glyde Stephen Panisset; William Charles Ross; Nevil Vincent Sidgwick, M.A., D.Sc.; Frank Sturdy Sinnatt; Robert Eley Blake Smith, B.Sc.; William Southworth; Francis Bernard Stead, B.A.; James Swain; Lyon Viccars Turner; Arthur James Webb, B.A.; Charles Edward Womersley.

Of the following papers, those marked * were read:—

*24. "*The Union of Hydrogen and Oxygen.*" By H. B. Baker.

Experiments have been made during the last ten years by H. B. Dixon, Victor Meyer, and by the author, with the object of seeing if the presence of moisture had any influence on the union of hydrogen and oxygen. These experiments have led to negative results. By the use of a new method of preparing very pure hydrogen and oxygen, namely, by the electrolysis of a solution of very pure barium hydroxide, the author has succeeded in preparing these gases so pure and dry that tubes containing them can be heated to redness without union of the gases, whilst tubes containing the undried gases, heated side by side with the dried tubes, readily explode. On the introduction of a small quantity of distilled water into the dried tubes, explosion follows at once. In gases which had been allowed to stand for two days only in contact with distilled phosphorus pentoxide, only a slow combination took place, so slowly that in one case ten minutes' heating in the flame of a Bunsen burner caused the union of only one-third of the partially dried gases. It seems therefore that water is not the only determining factor in the explosion of the heated mixture. In order to see if a still higher temperature would bring about the union, coils of very pure silver wire were heated by an electric current in the dried mixture, when it was found that the silver could be heated to its melting-point without causing union.

Experiments have also been made with the object of finding out if the moist gases were measurably dissociated whilst the dry gases were not. By a device of Professor Edward Morley's, used for another purpose, gases were sealed up in tubes in which a contraction of less than 1/7000th of their volume could be detected. The gases were dried before their introduction into the tubes by simply passing them through a phosphorus pentoxide tube. They were then left in contact with the pentoxide for six months. No contraction, even to the small extent above noted, could be observed. The gases tested thus

were hydrogen, oxygen, nitrogen, air, and a mixture of hydrogen and oxygen. These tubes were kept in a dark room during the whole period.

It was found that a moist mixture of hydrogen and oxygen united slowly in sunlight, which may perhaps account for some previous failures to get the gases dry enough not to unite. In the first series of experiments above described, the mixed gases were kept in darkness during their contact with the phosphorus pentoxide, lest the action of the drying agent should not be sufficiently rapid to cope with the water being produced by their union.

*25. "*Enzyme Action.*" By A. J. BROWN.

The author has already shown (*Trans.*, 1892, lxi., 380) that in alcoholic fermentation a *constant weight* of sugar is decomposed in unit time by a constant amount of yeast in solutions of equal volume containing different amounts of sugar, and has called attention to the fact that in this respect the action of fermentation differs essentially from that of inversion, which, according to C. O'Sullivan and Tompson (*Trans.*, 1890, lvii., 856), follows the law of mass action. At the time the author described his work, the phenomenon of fermentation was believed to be a life function inseparable from the living cell, and therefore it did not appear remarkable that the order of progression of its action should differ from that of inversion; but since Buchner has shown that fermentation, like inversion, is an enzyme action, this point of difference required further investigation, as the simple mass action of invertase demonstrated by O'Sullivan and Tompson is often regarded as typical of all forms of enzyme action.

The author has examined the action of invertase on cane-sugar, and finds that the velocity of its action differs essentially from that of a mass action, and *resembles that of fermentation*. The velocity of its action represented graphically approximates a *straight line* when not influenced by the accumulation of inversion products. The retarding influence of inversion products occasions the curve found by O'Sullivan and Tompson, and these authors were mistaken in regarding the curve as the logarithmic curve of mass action. In a true mass action the value K, derived from the expression $K = \frac{1}{\theta} \log \frac{1}{1-x}$, for any point of the action is a constant, but this is not the case in inversion changes in which the products of inversion accumulate. On the contrary, for such changes the value 2K is nearly constant when derived from the expression, recently suggested by Henri (*Comptes Rendus*, 1901, cxxxiii., 891), $2K = \frac{1}{\theta} \log \frac{1+x}{1-x}$,— an expression differing essentially from that representing the progress of a mass action.

But although the action of inversion does not follow the law of mass action, it cannot be independent of mass influence, and therefore the effect of mass during inversion change must be restricted by some other influence. This influence the author believes to be due to the existence of time as a factor in the complex changes which probably accompany inversion.

In any simple chemical change the influence of mass regulates the number of molecular *contacts* between acting and reacting molecules in unit time; but if a time factor enters into a molecular reaction there must be a point beyond which the number of molecular *changes* cannot increase owing to the restriction of time in the action, and this point will be determined by the relative frequency of molecular contact and the relative length of the time interval of molecular change. There is good reason to believe that during the inversion of sucrose, this sugar enters into molecular combination with invertase previous to change, which presupposes a complex change and the existence of a time factor of some magnitude. Under these conditions it therefore appears more probable that this factor limits the effect of mass action in inversion changes in solutions of ordinary concentration. But if this is so, there must be a point of dilution in cane-sugar solutions when invertase, acting in the dilute solutions, should exhibit an order of change in conformity with simple mass action. The author shows by direct experiment that this point is reached in a solution containing about 1 per cent of cane-sugar, so far confirming the conclusion that a time factor accompanying molecular change limits the action of inversion in all but very dilute solutions of cane-sugar.

The action of alcoholic fermentation follows the same order of progression as that of inversion, and the work of Kastle and Loevenhart (*Amer. Chem. Journ.*, 1900, xxiv., 491) indicates that the action of lipase progresses in the same manner—it therefore appears probable that both these enzyme actions are regulated, like inversion, by a time factor. With regard to the actions of such enzymes as diastase, glucase, and peptase there is not at present sufficient evidence on which to base an opinion.

*26. "*On the Velocity of Hydrolysis of Starch by Diastase with some Remarks on Enzyme Action.*" By H. T. BROWN and T. H. GLENDINNING.

The rate of change during the hydrolysis of starch by diastase does not conform to the simple logarithmic law of a unimolecular reaction. Throughout the hydrolysis there is a steady augmentation of the coefficient of velocity; in other words, within any given time interval there is more of the residual substance hydrolysed than there should be according to the logarithmic formula.

The results are in the wrong direction to be accounted for by a gradual accumulation of the products of change with a consequent tendency to chemical reversion, nor can they be explained by the intermediate products of change exhibiting a differential resistance to hydrolysis.

If the time curves expressing the rate of hydrolysis of a 3 per cent solution of soluble starch are critically examined, it is found that up to a hydrolysis of 30 to 40 per cent the amount of transformation is very nearly a linear function of the time. A further analysis of the curve shows that the remainder is approximately logarithmic.

In all essentials, the progress of the hydrolysis of starch coincides with that of the inversion of cane-sugar by invertase, as shown by Adrian Brown in the previous paper. The rate of hydrolysis also conforms closely with the empirical formula of Henri for the inversion of cane-sugar.

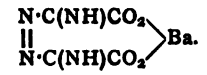
The authors are inclined to adopt a somewhat different explanation of the time curves of hydrolysis from that suggested by Adrian Brown, and one which does not postulate any difference in the time intervals between the successive stages of the reaction other than those due to variations in the respective masses of the reacting substances existing in unit volume.

This hypothesis has been fully elaborated, and an attempt is also made to link the phenomena of enzyme hydrolysis with those of acid hydrolysis, the water ions, or the active dissociated water molecules, being in both cases regarded as the true hydrolysts.

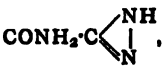
*27. "*Polymerisation Products from Diazoacetic Ester.*" By O. SILBERRAD, Ph.D.

In this research, which is a continuation of work already published (Hantzsch and Silberrad, *Ber.*, 1900, xxxiii., 58), the polymers of diazoacetamide are dealt with. The investigation has led to the discovery of a third series of polymeric products from diazoacetic ester, of which the most stable representative appears to be the amide. This compound, which was formerly know as "pseudodiazoacetamide, $C_3HN_4(NH)_2(CONH_2)_3$," is now shown to be imidoazoacetamide, $NH{:}C(CONH_2){\cdot}N{:}N{\cdot}C(CONH_2)NH$. Its constitution has been established chiefly by a comparative study of the action of reagents on this compound and on its isomerides in the other two series of polymeric derivatives of diazoacetamide, namely, the *C-* and *N-dihydrotetrazinedicarboxylamides*, the latter of which has been prepared for the first time. Of the reactions studied,

that with alkalis may be taken as characteristic. In the case of the *C*- and *N*-dihydrotetrazinedicarboxylamides saponification occurs in a perfectly normal manner; neither substance shows any inclination to form salts as imido azoacetamide does. In the presence of mercury, however, *N*-dihydrotetrazinedicarboxylamide forms a compound of such stability that prolonged boiling with concentrated sodium hydroxide entirely fails to saponify the amide. With pseudodiazoacetamide an imido-salt of the amide is first produced, as, for example, the ammonium salt $NH{:}C(CONH_2)N{:}N{\cdot}C(CONH_2)N(NH_4)$. It was the analysis of these salts which first showed the compound to be a dimolecular polymer of diazoacetamide. Secondary reactions subsequently occur thus (1) Baryta water on warming produces barium imidoazoacetate—

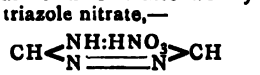


(2) The prolonged action of aqueous ammonia at ordinary temperatures gives rise to *iso*diazoacetamide,—

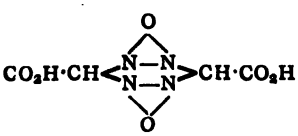


together with a small quantity of diazoacetamide. The constitution of the former was deduced chiefly from its action on benzaldehyde, with which it forms benzalazine, $N_2(CHC_6H_5)_2$. (3) Concentrated sodium hydroxide at 100° gives rise to bisdiazoacetic acid,—

The action of nitric oxide is also worthy of note as by its means either triazole nitrate,—



(a derivative of *N*-dihydrotetrazine), or bisazoxyacetic acid,—



(a derivative of *C*-dihydrotetrazine), can be obtained at will.

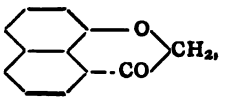
28. "*Condensation of Phenols with Esters of Unsaturated Acids.*" Part VII. By S. Ruhemann.

Benzo-1 : 4-pyrone (chromone) and its homologues have basic properties: they dissolve in hydrochloric acid to form hydrochlorides, and these solutions yield platinichlorides; but the salts are unstable, and are decomposed by water with re-formation of the benzopyrones.

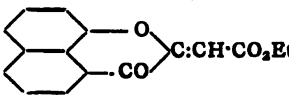
The author having been unable to obtain hydroxybenzo-1 : 4-pyrones from the dihydric phenols, has attempted to prepare them by starting from their monoethers, and from guaiacol and ethyl chlorofumarate has obtained ethyl guaiacoxyfumarate,—

a yellow oil, b. p. 212—213° (at 15 m.m.) and the corresponding acid (m. p. 138° with decomposition); on treatment of this acid with strong sulphuric acid a compound was obtained which is most probably methoxybenzo-1 : 4-pyronecarboxylic acid. This the author intends to transform into *O*-hydroxybenzo-1 : 4-pyrone.

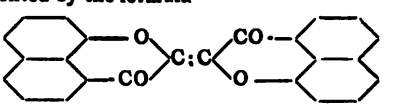
It was further shown that the action of ethyl chlorofumarate on α-naphthol differs from the behaviour of the ester towards β-naphthol, for whilst with the latter it yields ethyl β-naphthoxyfumarate, $C_{10}H_7O{\cdot}C(CO_2Et){:}CH{\cdot}CO_2Et$, a yellow, fluorescent oil, b. p. 240—242° (pressure 12 m.m.), which on hydrolysis is transformed into the corresponding acid, yellowish plates, m. p. 236° with decomposition, it reacts with α-naphthol to form in addition to a small quantity of ethyl α-naphthoxyfumarate, two substances which belong to a new class of compounds. One has the formula $C_{16}H_{12}O_4$, and crystallises in yellow needles, m. p. 146—147°; the other has the formula $C_{24}H_{12}O_4$, it forms orange needles which do not melt at 335°, are insoluble in the ordinary solvents, but dissolve in boiling nitrobenzene. The first compound is regarded as a derivative of the type—



called *naphtharone*, and as having the constitution—



ethyl naphtharonylacetate, whilst the compound $C_{24}H_{12}O_4$ is represented by the formula—



and termed *bisnaphtharonyl*.

Reasons for assigning these constitutions, and the description of some derivatives of these compounds, were given by the author.

29. "*The Chemical Change produced by the Immersion of Lead in Distilled Water.*" By F. Clowes, D.Sc.

The author finds when very pure lead and ordinary distilled water were employed that much of the lead which underwent change passed into solution most probably as hydroxide, and was removable to a large extent from solution by passage through filter-paper, from which it could again be entirely extracted by cold acetic acid. The compound of lead which remained undissolved by the water was found to have the formula $3PbCO_3,PbH_2O_2$.

In order to ascertain the respective parts played by atmospheric oxygen and carbon dioxide in solution, distilled water freed from dissolved gases by boiling was employed. When such water was placed in contact with lead in a vacuum or in an atmosphere of hydrogen, action only took place to the extent of 0·3 part of lead per million of water. When the water employed had been boiled in glass vessels and again exposed to air it never regained its full activity. This inhibitive effect varied with different glass vessels, being most marked in those kinds of glass which gave most dissolved matter to the water; contact of cold water and glass did not produce this effect, which was eliminated, however, in the experiments, the results of which are given below, by boiling the water employed in copper vessels.

The average results, expressed as percentages of lead in terms of the water employed, are shown in the following table:—

	24 hours.	48 hours.	72 hours.
Oxygen alone	0·013	0·023	0·029
Carbon dioxide alone	0·005	0·008	0·017
Equal volumes of carbon dioxide and oxygen	0·003	0·003	0·003
Eight volumes of oxygen to one volume of carbon dioxide ..	0·015	0·018	—

This shows clearly that oxygen is the principal and primary agent, and that carbon dioxide exerts a restraining action in proportion to the volume present. It was found that carbon dioxide acted similarly in preventing solution of litharge.

The first action of aërated water apparently consists in oxidation and formation of hydroxide, which is precipitated as hydroxycarbonate by the carbon dioxide. The action is prevented or retarded by the presence of carbon dioxide in the initial stages. Total immersion of the lead also

retards the action in presence of air, although the final result is the same whether the lead be wholly or only partially immersed in water.

Of the substances which prevent this action, sulphuric acid and soluble sulphates are most effective, soluble carbonates are less so, whilst calcium hydroxide is still less so, but when present in larger quantity actually promotes the action.

30. "*The Bases Contained in Scottish Shale Oil.*" Part I. By. F. C. Garrett and J. A. Smythe.

The authors are continuing the examination of the bases contained in the Broxburn shale oil, of which a short account has been already given (*Proc.*, 1900, xvi., 190), and have completed the investigation of the portion boiling below 164°. The following bases have been isolated and identified:—

Pyridine, b. p. 115—116° (in very small quantity).
α-Picoline, b. p. 129·5 (bar. 763 m.m.).
αγ-Dimethyl pyridine, b. p. 159—159·5°.
αβ'-Dimethyl pyridine, b. p. 154—155°.
αα'-Dimethyl pyridine, b. p. 142·5° (bar. 760 m.m.).
αγα'-Trimethyl pyridine, b. p. 170·5° (bar. 760 m.m.).

αβ'-Dimethyl pyridine is a colourless liquid with a strong pyridine-like odour; it gives a picrate, m. p. 151—152°; a gold compound, m. p. 156—157°; a platinichloride containing 2 molecules of water, and melting (when *anhydrous*) at 238° with decomposition; and a mercuri-chloride ($C_7H_9.HCl,6HgCl_2$) in very small, heavy crystals, m. p. 163°.

31. "*Note on 'Liquid Nitrogen Peroxide as a Solvent.'*" By P. F. Frankland, F.R.S., and R. C. Farmer, Ph.D.

In the authors' recent communication on "liquid nitrogen peroxide as a solvent" (*Trans.*, 1901, lxxix., 1356), they inadvertently omitted to mention the previous work of Bruni and Berti (*Gazz. Chim. Ital.*, 1900, xxx., [2], 151), of which they were not cognisant at the time.

It is fortunate that, whilst the two papers deal to some extent with the same subject, the experimental results do not to any great extent overlap, but rather supplement each other.

Bruni and Berti concerned themselves principally with cryoscopic measurements, and thus extended the still earlier work of Ramsay (*Trans.*, 1890, lvii., 590), whereas the authors have investigated the electric conductivity and ebullioscopic behaviour of the solutions of a considerable number of different substances in the liquid peroxide, besides making a somewhat extensive examination of the action of the latter on a number of other substances, organic and inorganic.

The cryoscopic and ebullioscopic determinations agree in showing that the liquid peroxide is in many cases a powerfully associating solvent, and it is noteworthy that Bruni and Berti found acetic acid to be associated to triple molecules at the freezing-point of the solvent, whilst the authors' measurements at the boiling-point of the latter only revealed an association to double molecules.

The authors desire herewith to express their regret that the important work of these authors had escaped their attention.

THE INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

The twenty-fourth Annual General Meeting of the Institute of Chemistry of Great Britain and Ireland was held at 30, Bloomsbury Square, London, W.C., on Monday, March 3rd, Professor J. Millar Thomson, LL.D., F.R.S., President, in the Chair.

The ordinary annual business of the Institute was transacted, and the President delivered an Address dealing with the work of the Institute during the past year.

The roll of Members has increased by 36, making the total of 1040 Fellows and Associates at present on the register, while there are 130 students in course of training at the Colleges and Institutions recognised by the Council of the Institute. The President remarked on the higher percentage of passes in the recent examinations, which he considered pointed to the fact that the thoroughness of the Examinations was more fully realised by those preparing for them.

He also mentioned the establishment of an Examination in Biological Chemistry, by means of which the Council hope to encourage analytical chemists to pursue the study of bacteriology, and thus be able to deal with chemical work involving bacteriological knowledge. The examination is specially suited for such as intend entering the practice of chemistry in its relationship to the chemistry and bacteriology of food, water, sewage, and effluents, and the practical applications of biological chemistry to such industries as brewing, tanning, &c. He called attention to the fact that the Institute held an Examination in Ireland in July last, and that the Council hope in future to arrange for a regular examining centre in Dublin.

During the past year the Council have ceased to recognise the Junior, Second Class, and Lower Grade Professional Preliminary Examinations of various examining bodies, and they have now under re-consideration the subjects which shall be compulsory to persons intending to proceed to the examinations of the Institute.

The Council also reported to the Members that during the past year they had addressed to the Local Government Board, and to various county and borough authorities, communications with reference to the combination of the two appointments of Medical Officer of Health and Public Analyst. It has been pointed out that a public analyst requires special training and experience in analytical chemistry far beyond that which is usually acquired by medical practitioners in the course of their professional education. Also, that where the appointments are proposed to be combined the selection of candidates for both positions is necessarily limited. The Council hold the opinion that under the provisions of the Sale of Food and Drugs Act of 1875, it was intended that the Medical Officer of Health and the Public Analyst for a borough or district should be two distinct persons, inasmuch as the Medical Officer is one of those officers empowered by the Act to submit suspected samples for analysis. The qualifications and examinations of the Institute are accepted by the Local Government Boards for public analytical appointments, and it is the desire of the Council that no lower standard of chemical training and experience than that prescribed by the Institute of Chemistry should be accepted for such important and responsible public appointments as those of Public Analysts under the Sale of Food and Drugs Act.

CORRESPONDENCE.

REGULATIONS FOR MILK AND CREAM.

To the Editor of the Chemical News.

Sir,—In the recommendations of the Departmental Committee appointed by the Board of Agriculture to inquire into and report upon the desirability of Regulations for Milk and Cream, 1901, under (*c*) it is said—"In calculating the percentage amount of admixed water the analyst shall have regard to the above-named limit of 8·5 per cent of non-fatty milk-solids, and shall further take into account the extent to which the milk-fat may exceed 3·25 per cent."

A case came before one of us many years ago of a milk which looked rich and actually contained over 4 per cent of fat, but which, calculated on the solids other than fat, showed 40 per cent of added water. Some years afterwards this curious case was explained by a country dairy-

man, who said it was quite simple, as the supplier of the milk had skimmed the evening's milk and added the cream to next morning's, with as much water as there was of skimmed milk, which he then had for his calves.

In the recommendations referred to 12 per cent total solids is taken as the minimum, of which 3·25 per cent must be fat; but suppose that a milk had been manipulated in the way shown, and that it contained 12 per cent of total solids, 4 per cent of which was fat, it might still be watered to the extent of 16 per cent.—We are, &c.,

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CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxiv., No. 7, February 17, 1902.

Action of Potassium Hydride on Ethyl Iodide and Methyl Chloride. New Preparation of Ethane and Methane.—Henri Moissan.—Potassium hydride reacts on ethyl iodide and methyl chloride by fixing hydrogen on to these compounds and liberating the iodine or the chlorine in the form of a binary compound, the alkaline iodide or chloride. This reaction takes place in a sealed tube between 180° and 200° perfectly regularly, without the deposition of carbon and without the liberation of iodine or chlorine.

Conductibility of Liquid Dielectrics under the Influence of Radium Radiation and Röntgen Rays.—P. Curie.—The author proves that the rays emitted by radium and also Röntgen rays act on liquid dielectrics in the same way as they do on air, communicating to them a certain electric conductibility. The apparatus by which this is experimentally demonstrated is described and drawn. In the case of both forms of radiation results of the same order of greatness were obtained. The liquids examined were—Carbon disulphide, petrol ether, amylene, chloride of carbon, benzine, liquid air, and vaseline oil.

Praseodymium Chloride.—Camille Matignon.—The author first determines the principal physical constants of praseodymium chloride by a series of experiments on its progressive desiccation in a current of hydrochloric acid gas. He next proves the existence of a new hydrate of the chloride, $PrCl_3.H_2O$, and finally gives a simple method of preparation of the anhydrous chloride. The chloride of praseodymium and neodymium when examined together present no marked difference in their properties which could be utilised for fractionation in order to separate them.

MISCELLANEOUS.

Some Platino carbon Compounds.—W. Prandtl and K. A. Hofmann.—A solution of chloroplatinic acid in oxide of mesityl is left, *in vacuo*, over caustic soda; after about a fortnight a crystalline mass is formed, blackened by platiniferous products, and hydrochloric acid is given off. The crystals are washed with a mixture of acetone and ether, and re-crystallised in boiling acetone. Pale yellow needles are formed, very soluble in acetone, slightly soluble in ether, and soluble in cold water; this latter solution decomposes spontaneously. The composition of the product is $PtCl_2.C_6H_{10}O$; it is identical with the *acechloroplatinum*, obtained by Zeise, by reacting with $PtCl_4$ on acetone (*Journ. f. Prakt. Ch.*, vol. xx., p. 193). If we boil $PtCl_6H_2$ with chloroform for several days, hydrochloric acid is given off, and a thick brown residue is left, covered with a yellowish liquid. This latter was evaporated, then taken up with ether, the solution was filtered and evaporated, and again taken up with ether; then, on evaporation, we obtained pale yellow needles, which were crystallised in warm toluene. The substance answers to the formula $PtCl_2.C_2H_4$; it is not deliquescent, is stable when exposed to light; when heated to about 140° it commences to decompose.—*Berichte*, vol. xxxiii., p. 2981.

The Ketones of Anthracene.—E. Lippmann and P. Keppich.—It is known that the property of a quinonic oxygen of reacting with hydrochlorate of hydroxylamine is diminished when the two atoms of hydrogen in the ortho-position are replaced by alcoholic residues or by the halogens; it is also known that tetra- and penta-methylphloroglucine are indifferent with regard to phenylhydrazine and hydroxylamine, and that the benzoic and meso-anthracene carbonic acids substituted in ortho-, do not give, according to Victor Meyer's law, any ether by the action of alcohol and hydrochloric acid. Having regard to all these observations, it might be supposed that when we replace an atom of hydrogen placed in the meso-position by an acid residue, in the molecule of anthracene, the formation of hydrogenes and oximes would be hindered by the neighbouring position of the two atoms of carbon in the lateral benzenic radical. The researches of the authors on this subject have confirmed this supposition. They describe their method for the preparation of anthraphenone; they then describe its oxidation and reduction. They have also prepared nitro-anthraphenone, as well as tribenzoylanthracene and its tetrahydrogenised derivative. —*Berichte*, vol. xxxiii., p. 3086.

MEETINGS FOR THE WEEK.

MONDAY, 17th.—Society of Arts, 8. (Cantor Lectures). "Photography applied to Illustration and Printing," by J. D. Geddes, F.R.P.S.

TUESDAY, 18th.—Royal Institution, 3. "Recent Researches on Protective Resemblance, Warning Colours, and Mimicry in Insects," by Prof. E. B. Poulton, M.A., Hon. LL.D., D.Sc., F.R.S.

WEDNESDAY, 19th.—Society of Arts, 8. "Electric Traction—London's Tubes, Trams, and Trains," by J. Clifton Robinson, Assoc.Inst.C.E., M.I.E.E.

—— Chemical, 5.30. "The Absorption-spectra of Metallic Nitrates" (Part I.), by W. N. Hartley. "A Method of Determining the Ratio of Distribution of a Base between two Acids," by H. M. Dawson and F. E. Grant. "On the Molecular Complexity of Acetic Acid in Chloroform Solution," by H. M. Dawson. "On the Existence of Polyiodides in Nitrobenzene Solution," by H. M. Dawson and R. Gawler. "Nitrogen Chlorides containing the Propionyl Groupe," by F. D. Chattaway. "Derivatives of α-Aminocamphoroxime," by A. Lapworth and A. W. Harvey. "Preparation of Sulphamide from Ammonium Amidosulphite," by E. Divers and M. Ogawa. "Hypoiodous Acid," by R. L. Taylor.

—— Microscopical, 8. Exhibition of Foraminifera, by A. Earland.

THURSDAY, 20th.—Royal Institution, 3. "Caricature in and out of Parliament," by E. T. Reed.

FRIDAY, 21st.—Royal Institution, 9. "Recent Developments in Colouring-matters" (in English), by Geheimrath Professor Otto N. Witt, Ph.D., F.C.S., of Berlin.

SATURDAY, 22nd.—Royal Institution, 3. "Some Electrical Developments," by Lord Rayleigh, F.R.S., &c.

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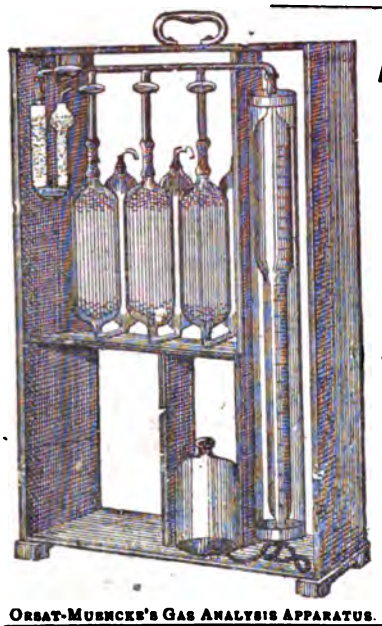

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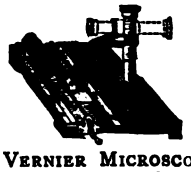

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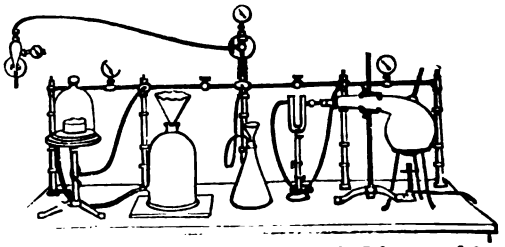

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THE CHEMICAL NEWS.

VOL. LXXXV., No. 2208.

NOTE ON THE ANOMALOUS DISPERSION OF SODIUM VAPOUR.*

By W. H. JULIUS, Professor of Physics in the University of Utrecht.

THE importance of the study of anomalous dispersion in gases, both for testing dispersion theories, and for investigating the connection of the subject with solar physics,† has induced Professor R. W. Wood to publish a highly interesting paper on the anomalous dispersion of sodium vapour.‡

In most of his experiments, Wood deals with a much denser vapour than I had studied; this may be the reason that he believes he finds a difference between his views and mine about a point on which, in fact, we do agree.

On p. 160 (*loc. cit.*). Wood describes the spectrum of electric light that has passed through a tube containing sodium vapour of increasing density, and how in a few seconds the vapour becomes so dense that total absorption of all the light between the D-lines occurs. Then he says:—"Julius expresses the opinion that this disappearance of the light between the lines is only a result of the strong dispersion"; and he is of a different opinion. This, however, proves, I fear, that he really did not read my paper rightly, as he suspected might be the case (*loc. cit.*, p. 169).

Indeed, speaking of the difference in the result obtained by Becquerel and by myself in studying the dispersion caused by a sodium flame, I suggested (*Roy. Acad. Amsterdam Proc.*, p. 578) that "perhaps Becquerel's flame contained more sodium than mine"; for I felt sure, that in a sufficiently dense vapour the absorption would extend over a broader region of the spectrum. Some lines further, alluding to the absence, in the spectrum, of the light that is strongly deflected by anomalous dispersion and thus falls outside the spectroscope, I said:—"Here, then, we have a case where the absorption spectrum of a vapour exhibits broad bands not deserving the name of absorption bands"; and then . . . . "It would be worth while investigating in how far the anomalous dispersion can have influenced cases, in which broadening or reversal of absorption-lines have been observed."

So it is quite clear that I did not deny the possibility of getting broad bands by absorption. I have never expressed the opinion, that the disappearance of the light between the D-lines in the absorption spectrum of dense sodium vapour is only a result of the strong dispersion; but I warned against always ascribing the observed dark bands to absorption only.

Wood's recent researches are very important as a contribution to our knowledge of dispersion in general. For the present their bearing on the spectral phenomena exhibited by the light from the chromosphere and from sun-spots, seems not to be so direct, because, most probably, the density of the vapours is much less in the solar atmosphere than in the dispersion tubes used by Wood in his brilliant experiments.

An Important Source of Error during Researches on Diastase.—M. Ernow Pozzi-Escot.—The author's researches on diastase lead him to the conclusion that a certain number of published works on diastases and their localisation in special cells lose some of their value when certain facts with regard to colour reactions of diastases are taken into consideration.—*C. R.*, cxxxiv., No. 8.

* A Paper read before the Royal Society, February 20, 1902.

† W. H. Julius, "Solar Phenomena Considered in Connection with Anomalous Dispersion of Light," *Roy. Acad. Amsterdam Proc.*, vol ii., p. 575; *Astron. Nachr.*, 3672.

‡ R. W. Wood, "The Anomalous Dispersion of Sodium Vapour," *Roy. Soc. Proc*, vol lxix., p 157.

VALENCY OF OXYGEN AND THE HALOGENS.

By H. STANLEY.

KEKULÉ (*Lehrbuch*, i., 168) propounded the two principles on which our present theory is based, viz., that carbon is a tetravalent element, and that its atoms have the property of combining among themselves. This latter condition partly accounts for the immense number of carbon compounds. Tilden says:—". . . Even supposing all the organic compounds completely burnt up, there will still remain a considerable quantity of oxygen. Oxygen is entirely unmatched among the rest of the elements, both as regards the number and varied character of its compounds."

Does oxygen present a case at all analogous to that of carbon?

Heynes has divided the elements into two classes:—(1) Those whose valency is a single number; (2) those whose valency may be represented by a double number.

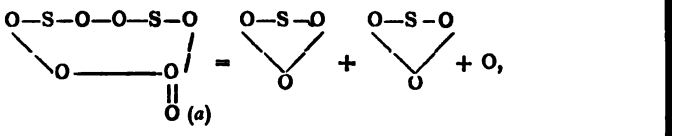
In the latter class he places oxygen and the halogens. The polyatomicity of these elements seems to come into play chiefly when the atoms of these elements unite among themselves.

Peroxides are divided into two classes. To the first of these belong such bodies as Na_2O_2 or $\begin{matrix}Na—O\\ |\\ Na—O\end{matrix}$; and to the second belong the peroxides of barium and hydrogen—

(1) $Ba{=}O{=}O_{(a)}$, (2) $\begin{matrix}H\\ H\end{matrix}\!\!>\!O{=}O_{(a)}$.

The formula of sulphur dioxide may be written $S\begin{matrix}O\\ |\\ O\end{matrix}$ and, in analogy with this, we may write ozone as the corresponding oxygen compound, $O\begin{matrix}O\\ |\\ O\end{matrix}$. This is a symmetrical arrangement, and represents each O atom standing in the same relation to the rest of the molecule. The fact that ozone readily decomposes when heated lends strong support to such a formula as $O{=}O{=}O_{(a)\ (a)}$, where one of the atoms (*a*) is disengaged on heating, leaving a molecule of oxygen, O=O. If we compare ozone and hydrogen peroxide, we find that they act both as reducing and as oxidising agents, and the "available" O atom is in each case united to a second O atom, which resumes its normal valency on the breaking down of the molecule.

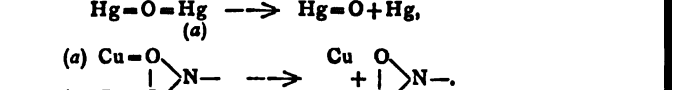
The decomposition of sulphuric anhydride may be expressed—



and many other similar reactions in which the quadrivalence of oxygen may be assumed.

Apparently, however, this high valency of oxygen is not confined to cases of combination between atoms of its own kind, but also when united to other divalent atoms. Thus, for example, take the cases of certain suboxides which decompose on heating, leaving a higher oxide and the metal:—

$$Hg{=}O{=}Hg_{(a)} \longrightarrow Hg{=}O + Hg,$$

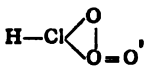


(Copper nitroxyl).

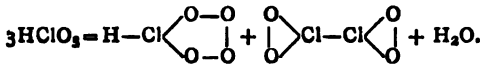
The increasing stability of the series of oxy-acids of

chlorine is probably connected with the quadrivalence of oxygen.

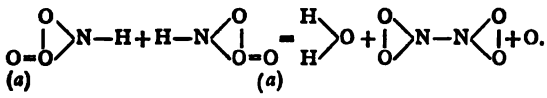
Suppose we write the formula of chloric acid—



its decomposition into perchloric acid and chlorine peroxide may be represented thus :—



Compare with this the decomposition of nitric acid—



(a) (a)

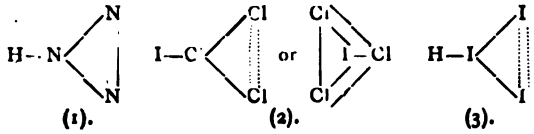
Concentrated HNO_3 boils at 86° and partially decomposes into water, N_2O_4, and oxygen).

Perchloric acid and its salts are more stable than the chlorates and yield no ClO_2 on heating, but at once evolve oxygen, leaving a chloride.

Oxygen in its dyad capacity in $HClO_4$ produces a more stable compound than the oxygen in $HClO_3$, one of the atoms exhibiting quadrivalence.

Baeyer has, however, recently pointed out that ethyl peroxide may be readily reduced to alcohol and inclines to the opinion that hydrogen peroxide must be represented as HO.OH rather than as $O:OH_2$. Collie had previously suggested the formula $Et_2=O=O$ for ethyl peroxide.

The halogens in a tervalent capacity show a marked analogy with nitrogen. Thus we have hydrazoic acid (HN_3), iodine trichloride (ICl_3); and the recent work of Dawson (*Trans. Chem. Soc.*, 1901, 238) plainly points to the existence of such compounds as KI_3 derived from an acid HI_3. Thus we have—



(1). (2). (3).

Many cases in which the halogens are apparently tervalent are known, especially amongst double compounds.

Stortenbeker (*Zeit. Physikal. Chem.*, 1892, x., 183) finds that ICl_3 dissociates into ICl and Cl_2, the molecular depression (in solutions in glacial acetic acid) decreasing with rise of concentration. This consideration would lend support to the first of the two formulæ given for iodine trichloride above, which then presents analogy with HN_3 and the (as yet) hypothetical acid HI_3 (*cf.* the decomposition of $HClO_4$).

The existence of complex molecules of hydrofluoric acid is readily explained if we accept the tervalent character of fluorine, as is also the constitution of the double compounds of the general formula $MF_n{\cdot}n(NH_4)F$ (Poulenc, *Comptes Rendus*, 1892, cxiv., 1426).

It thus seems that oxygen and the halogens (sulphur and possibly other elements) exhibit a valency of a higher order than is normal to them, and that this increase takes place most readily when combinations among atoms of a like kind take place; that the compounds formed by this means are of a lower order of stability than those in which the normal valency is exerted, and that scission takes place in such compounds between the rest of the molecule and that element which is exerting this higher valency. On decomposition taking place, that element resumes its normal atom-fixing power, unless the conditions are such as to cause this power to be still exerted.

Merchant Venturers' Technical College,
Bristol.

THE EFFECT OF SEA-WATER ON MUNTZ'S METAL SHEATHING.

By ERNEST A. LEWIS.

MUNTZ'S metal is a well-known alloy containing from 60 to 62 per cent copper and from 38 to 40 per cent zinc, which has been used for over fifty years for covering the bottoms of wooden ships, and covering piles in harbours, docks, &c. The usual theory assigned for the application of this alloy is that the zinc is slowly and uniformly corroded over the surface by exposure to sea-water, whereby the attachment of barnacles, &c., is prevented.

I have recently had to examine a large number of samples of sheathing which had worn away much too rapidly; some samples had been on piles for only twelve or eighteen months, instead of seven or eight years. The metal in every case became brittle. This peculiar behaviour of the metal is more remarkable because there has been no change in the mode of manufacture, and the metal has to stand bending and tensile tests before leaving the manufactory.

The following are analyses of good sheathing which had been in use some time, and did not show signs of deterioration :—

No. 1.

	Per cent.
Copper	61·02
Tin	0·29
Lead	0·567
Iron	0·11
Zinc	38·013
	100·000

No. 2.

	Per cent.
Copper	61·66
Tin	0·414
Lead	0·94
Iron	0·288
Zinc	36·698
	100·000

No. 3.

	Per cent.
Copper	60 06
Tin	0·431
Lead	0 922
Iron	0 262
Zinc	38·325
	100·000

No. 4.

	Per cent.
Copper	61·388
Tin	0·511
Lead	0·436
Iron	0·027
Manganese	nil
Arsenic	0·009
Zinc	37 629
	100·000

The following are analyses of bad sheathing, which had rapidly deteriorated :—

No. 1*a*.

	Per cent.
Copper	64·704
Tin	0·004
Lead	0·506
Iron	0·055
Arsenic	0·008
Zinc	34·723
	100·000

No. 2*a*.

	Per cent.
Copper	66·275
Tin	traces
Lead	0·346
Iron	0·05
Nickel	0·057
Arsenic	0·006
Zinc	33·266
	100·000

No. 3*a*.

	Per cent.
Copper	62·006
Tin	nil
Lead	0·357
Iron	0·057
Nickel	nil
Arsenic	0·016
Zinc	37·564
	100·000

No. 4*a*.

	Per cent.
Copper	96·562
Tin	0·021
Lead	0·099
Iron	0·01
Zinc	3·308
	100·000

No. 5*a*.

	Per cent.
Copper	92·19
Tin	traces
Lead	0·19
Iron	0·023
Nickel	0·098
Arsenic	0·007
Zinc	7·492
	100·000

It will be noticed that, in the samples of good sheathing, the percentages of tin and iron are considerably more than in the bad sheathing. I made a sample of Muntz's metal with chemically pure copper and zinc, and had it rolled and annealed exactly as the metal is manufactured. I then put it in sea-water in an open beaker for a month; the surface became rough, and the sea-water dissolved away some of the zinc.

I also made samples of Muntz's metal containing known amounts of impurity and treated them like the chemically pure metal.

No.	Impurity added.	Appearance after one month.
1.	0·2 per cent tin	The surface was smooth.
2.	0·2 ,, arsenic	,, ,, ,,
3.	0·2 ,, antimony	The surface was smooth, but the antimony made the metal brittle.
4.	0·2 ,, iron	The surface was fairly smooth.
5.	0·2 ,, nickel	The surface was fairly smooth.
6.	0·2 ,, manganese	The surface was smooth.

Muntz's metal consists of two compounds; the one freezes at 894° C., and the other at 460° C. The one freezing at 894° C. is a compound of copper with Cu=Zn, the other is a eutectic alloy of "Cu=Zn+*x*Zn." When Muntz's metal is cast, the "*x*Cu+Cu=Zn" compound solidifies first, and then the "Cu=Zn+*x*Zn" eutectic solidifies; this Cu=Zn eutectic will contain all the impurities originally present in the copper and zinc.

When Muntz's metal is re-heated and rolled, the Cu=Zn eutectic melts and the "*x*Cu+Cu=Zn" compound is raised to the heat at which it is malleable. When the metal is passed through the rolls, these crystals of "*x*Cu+Cu=Zn" are flattened, and the Cu=Zn eutectic is forced to the edges of the crystals of "*x*Cu+Cu=Zn." When the "*x*Cu+Cu=Zn" cools, it breaks up into very much smaller crystals, which are probably Cu_2Zn.

I have already shown that chemically pure Muntz's metal is easily attacked by sea-water, and that Muntz's metal containing 0·2 per cent tin, arsenic, or manganese is able to resist corrosion much better than the pure metal, and also that Muntz's metal containing 0·2 per cent of iron or nickel is able to resist the corrosive action of the sea-water better than pure Muntz's metal, but not so well as the metal containing 0·2 per cent of tin, arsenic, or manganese.

Lead does not appear to have any influence in making Muntz's metal resist corrosion; every sample of sheathing I have examined has contained from 0·1 to 1 per cent of lead.

Owing to improvements in the manufacture of copper and zinc, these metals are now sold considerably purer than they were years ago; it is now easy to obtain copper of 99·9 per cent purity, and the double distilled spelters contain but traces of impurity.

I believe that Muntz's metal sheathing resists corrosion owing to it containing small amounts of metals as impurities, and that if it does not contain a small proportion of tin, arsenic, iron, manganese, or nickel, it is of no value for ship's sheathing and similar purposes.

In pure Muntz's metal the eutectic alloy of Cu=Zn is comparatively easily attacked by sea-water; the zinc in the Cu=Zn eutectic dissolves away, leaving thin layers of copper, which then forms a galvanic couple with the "*x*Cu+Cu=Zn" compound and rapidly dissolves away the zinc, leaving finally a brittle mass of copper (see analyses No. 4*a* and No. 5*a* above).

In a Muntz's metal containing small percentages of tin, arsenic, iron, manganese, or nickel, these metals dissolve in the Cu=Zn eutectic and enable it to resist corrosion for a much longer time, and until the zinc in the Cu=Zn eutectic is dissolved away, the zinc in the "*x*Cu+Cu=Zn" compound is not acted upon.

It has been shown that, in manganese-brasses, stereometal, Aich's metal, and similar alloys (see "Fourth Report to the Alloys Research Committee of the Institution of Mechanical Engineers"), the iron or manganese added to them dissolve in the lower eutectic and raise its melting-point nearly to the melting-point of the "*x*Cu+Cu=Zn" compound. These metals also have the property of resisting corrosion much better than similar alloys without the iron or manganese, because the Cu=Zn eutectic, when it contains iron or manganese, is able to resist the action of the corrosive liquid for a greater time, thus preventing the formation of minute galvanic couples among the microscopic compounds of the alloys.

ON PYRITE AND MARCASITE.*

By H. N. STOKES.

(Continued from p. 121).

XII. Constitution of Pyrite and Marcasite; Action of Cupric Salts.

The importance to chemical geology of an experimental study of the action of copper salts on pyrite and marcasite is self-evident. The following experiments were made, however, with the object of ascertaining whether the method of A. P. Brown (*Proc. Am. Phil. Soc.*, 1894, xxxiii., 225; Chem. News, 1895, lxxi., 179)—namely, heating with neutral copper sulphate solution at 200°)—could be used to distinguish these minerals, and incidentally, of testing his hypothesis that the iron in marcasite is wholly ferrous, while that of pyrite is four-fifths ferric. I have therefore confined myself to the conditions of temperature and concentration observed by him.

* From the *Bulletin of the U.S. Geological Survey*, No. 186.

Brown, in his published experiments, has limited himself to titrating the ferrous iron in the resulting solution, while no determination was made of any ferric iron which might be present either in the solution or in the precipitate. As neutral ferric sulphate is practically completely precipitated as basic salt on heating its solution, and as any ferric salt remaining in solution would inevitably be reduced by the sulphides present, it seems reasonably clear that a single experiment with pyrite, in which only the ferrous salt is determined, in which no account is taken of the ferric salt supposed to be formed, and in which no clear evidence of complete decomposition of the mineral is given, can not have great weight in establishing an important hypothesis. While not questioning the correctness of Brown's single observation so far as it goes, I have been wholly unable to obtain the same results.

In each experiment cited below the material was finely ground, carefully freed from oxidation products in the manner employed in my oxidation experiments (p. 63), and about 0·2 grm. heated with 50 c.m.³ of 10 per cent iron-free $CuSO_4.5H_2O$ solution which had been freed from air by boiling out and cooling in carbon dioxide. The tubes were filled with carbon dioxide, carefully sealed in a current of the same gas, and heated for varying times at 200° C. The filtration and titration with permanganate of the resulting solution were also made in an atmosphere of carbon dioxide, and the end-reaction, which is not very sharp, was determined by comparison with a copper sulphate solution of about the same strength.

Experiments with Cupric Sulphate and Pyrite.

Experiment 1.—Pyrite No. 4. Oxidation coefficient, 60·3. Time of heating, ten hours.

Pyrite taken	0·2024
Ferrous iron in solution	0·0305
Total iron in solution	0·0314
Total iron in pyrite used	0·0944

The solution therefore contains 33·3 per cent of the iron, practically all in the ferrous condition, against 20 per cent as required by Brown's hypothesis. The precipitate was red-brown. In a separate experiment the red substance was found to be left behind on oxidising the sulphide with dilute nitric acid, and proved to be ferric oxide or a very basic sulphate. It is easily soluble in hot dilute hydrochloric acid, and can thus be separated from the sulphide and determined.

Experiment 2.—Pyrite No. 5. Oxidation coefficient, 60·8. Time of heating, eleven hours.

Pyrite taken	0·2028
Ferrous iron in solution	0·0510
Total iron in solution	Same
Total iron in pyrite used.. ..	0·0946

The solution contains 53·9 per cent of the iron, all in the ferrous state. The ferric oxide in the red-brown precipitate was extracted by boiling ten minutes with dilute hydrochloric acid in a current of carbon dioxide, whereby some copper also dissolved. The ferric iron thus determined was 0·0272 grm. The final residue, oxidised with aqua regia, gave much iron, which must have come from undecomposed (or imperfectly decomposed) pyrite. From the sum of the two irons above determined it appears that 82·7 per cent of the pyrite was decomposed, and that this portion gave—

62·5 per cent ferrous iron.
34·8 per cent ferric iron.

Required by Brown's hypothesis, 20 per cent ferrous iron.

Experiment 3.—Pyrite No. 5. Time of heating, seven hours.

Pyrite used..	0·2027
Ferrous iron in solution	0·0527
Total iron in solution	0·0523
Total iron in pyrite used.. ..	0·0945

The solution therefore contains 55·3 per cent of the iron entirely as ferrous salt. The ferric oxide, extracted as above from the red brown precipitate, was equivalent to 0·0225 grm. iron. The final residue contained much iron. From these data it follows that 79·1 per cent of the pyrite was decomposed, the dissolved portion giving—

69·9 per cent ferrous iron.
30·1 per cent ferric iron.

Required by Brown's hypothesis, 20 per cent ferrous iron.

Experiments with Cupric Sulphate and Marcasite.

Experiment 4.—Marcasite No. 10. Oxidation coefficient, 17·4. Time of heating, seven hours.

Marcasite used	0·2062
Ferrous iron in solution	0·0559
Total iron in solution	0·0562
Total iron in marcasite used ..	0·0961

After extracting the red-brown precipitate as above, the residual sulphide was found to be free from iron, indicating complete decomposition of the marcasite. From these data it follows that the marcasite yielded—

58·2 per cent ferrous iron.
41·8 per cent ferric iron.

Required by Brown's hypothesis, 100 per cent ferrous iron.

Experiment 5.—Marcasite No. 10. Time of heating, fourteen hours.

Marcasite used	0·2096
Ferrous iron in solution	0·0651
Total iron in solution	Same
Ferric iron from precipitate ..	0·0318
Total iron in marcasite used ..	0·0977

The precipitate further yielded to hydrochloric acid 0·0626 grm. copper, which was present either as cuprous oxide or as metallic copper, while the final residue was free from iron, showing that complete decomposition had been effected. The above data give for this experiment—

66·6 per cent ferrous iron.
33·4 per cent ferric iron.

Required by Brown's hypothesis, 100 per cent ferrous iron. The copper sulphide remaining after extraction with hydrochloric acid contained 0·2309 grm. copper, from which it follows that two atoms of copper are precipitated as sulphide from each atom of iron dissolved. This necessarily implies a reduction of the copper to the cuprous condition, and a corresponding oxidation of a portion of the ferrous salt or of the sulphur, or of both. That cupric salts can accomplish both of these results is shown in the two following experiments.

(To be continued).

Royal Institution.—The following are the lecture arrangements at the Royal Institution, after Easter:—Dr. Allan Macfadyen, three lectures on "Recent Methods and Results in Biological Inquiry"; Prof. F. York Powell, three lectures on "English Kings and Kingship"; Professor Karl Pearson, three lectures on "The Laws of Heredity, with Special Reference to Man" (the Tyndall Lectures); Professor Dewar, three lectures on "The Oxygen Group of Elements"; Dr. A. Smith Woodward, three lectures on "Recent Geological Discoveries; Mr. M. H. Spielman, three lectures on "Contemporary British Sculpture"; Mr. W. H. Cummings, three lectures on "British National Song" (with musical illustrations); Professor Walter Raleigh, three lectures on "Poets and Poetry"; and Professor Brander Matthews, three lectures on "The Development of the English Drama—(1) The Art of the Dramatist; (2) The Drama of The Middle Ages; (3) The Drama under Elizabeth." The Friday Evening Meetings will commence on April 11th, when Professor Dewar will deliver a discourse on "Problems of the Atmosphere." Succeeding Friday Evening Discourses will be delivered by Sir John H. A. Macdonald, Dr. J. Mackenzie Davidson, Sir Robert Ball, Sir Benjamin Baker, Professor A. E. Tutton, and other gentlemen.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, March 6th, 1902.

Dr. DIVERS, F.R.S., Vice-President, in the Chair.

MESSRS. W. G. Black, L. V. Turner, and H. Lucas were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. James Samuel Ashe, The Adelaide Hospital, Dublin; Arthur James Cooke, 167, Richmond Road, Hackney, N.E.; Henry Arthur Dobson, Paisley Street, Orange, N.S.W.; James Kewley, King's College, Cambridge; Alexander Macknight, Brucefield, Whitburn, N.B.; William Harrison Martindale, 10, New Cavendish Street, W.; John Wicliffe Peck, 28, West Ella Road, Harlesden, N.W.

The list of names of those recommended for election as Officers and Members of Council was read.

Of the following papers, those marked * were read:—

*32. "*The Slow Oxidation of Methane at Low Temperatures.*" By W. A. BONE and R. V. WHEELER.

The authors have studied the slow oxidation of methane at temperatures between 300° and 400° in a supply of oxygen just sufficient to burn the carbon to carbon monoxide. The method adopted consisted in sealing mixtures of two volumes of methane and one volume of oxygen in bulbs of boro-silicate glass, which were maintained at constant temperatures of 300°, 325°, 350°, and 400° for periods of time varying from one day to three weeks. The bulbs were afterwards examined and a careful analysis of the resulting mixture made.

The results indicate—(1) That the disappearance of the oxygen is accompanied by a proportionate diminution in the volume of the cooled products, due to formation of water; (2) that at no stage of the process is either free hydrogen or free carbon liberated; (3) that a portion only of the methane is burnt, the carbon of which appears as carbon monoxide or dioxide, and the whole of the hydrogen as water; (4) that no other products can be detected at any stage of the oxidation.

Since the interaction of methane and oxygen at these temperatures is a "surface" phenomenon, it is impossible to measure the relative velocities of the reaction at different temperatures, but evidence was obtained that the velocity increases with rising temperature. Thus at 300°, where the velocity is just appreciable, the disappearance of oxygen and formation of water can only be detected after the lapse of one or two weeks. At 400°, on the other hand, the whole of the oxygen always disappeared within a single day, and at 350°, in some instances, in three days.

Experiments have shown that similar bulbs containing electrolytic gas can be maintained at 350° for a week and at 400° for three days, without any visible formation of water occurring; at 400°, in one instance out of three, a marked formation of water took place in a week; in the other two bulbs, however, no change could be detected.

A study of the following possible secondary reactions at 325°, 350°, and 400°, namely (1) $CO+H_2O \rightleftarrows CO_2+H_2$, and (2) $2CO+O_2=2CO_2$, indicates that no complication (moist) arising from them enters into the methane-oxygen experiments. Further, it has been proved that the following substances do not react at 350—400°, namely, (1) methane and steam, (2) methane and carbon dioxide, (3) carbon and steam.

The authors therefore conclude that the first stage of the oxidation of methane at these temperatures does not consist of a "*selective combustion*" of either hydrogen or carbon, but in a *simultaneous oxidation of carbon and hydrogen*, as represented by the equation $2CH_4+3O_2=2CO+4H_2O$.

The occurrence of carbon dioxide in the products (and in many cases as much as one-third to one-half of the carbon burnt appears as the dioxide) cannot be explained on the supposition that carbon monoxide liberated in the primary oxidation is subsequently *gradually* oxidised through the agency of steam and oxygen, or steam alone. The more probable explanation is that the carbon monoxide and steam molecules which simultaneously come into being in the primary oxidation are, *at the moment of formation*, in a very labile and reactive condition, and this would favour the rapid exchange of oxygen in the system $\begin{matrix}CO\\CO\end{matrix}\Big|\begin{matrix}OH_2\\OH_2\end{matrix}\Big|O_2$. The carbon dioxide is mainly, if not entirely, produced during this short "labile period."

*33. "*Isomeric Additive Compounds of Dibenzylketone and Deoxybenzoin with Benzal-p-toluidine, m-Nitrobenzalaniline, and Benzal-m-nitraniline.*" Part III. By F. E. FRANCIS, B.Sc., Ph.D.

The additive compounds formed with dibenzylketone and deoxybenzoin with benzal-*p*-toluidine were unstable, and consequently unsatisfactory for confirming the existence of the so-called α-, β-, and γ-modifications. In the case of dibenzylketone and *m*-nitrobenzalaniline the β-modification obtained by re-crystallisation of the α-form from benzene containing traces of piperidine melted 31° higher, and with dibenzylketone and benzal-*m*-nitraniline 43° higher than the α-additive product; in the latter case, there was also a distinct change in the solubility and appearance of the β-form. These were considered satisfactory confirmations of the individuality of the β-modification, because in cases previously investigated the rise in melting-point was never greater than 10—12°, and since the β-modifications were transformed into the α- by means of heat, this small difference in melting-point was not very satisfactory. α-Dibenzylketone benzal-*m*-nitraniline on re-crystallisation from benzene containing traces of sodium ethoxide appears to be converted into the γ-modification, and the melting-point rises from 134° to 182·3°, but this was the only case observed. Substances of much higher melting-point and smaller solubility, and apparently belonging to neither α-, β-, or γ-modifications, were obtained in the case of deoxybenzoin and benzal-*p*-toluidine, deoxybenzoin and benzal-*m*-nitraniline, and dibenzylketone and *m*-nitrobenzalaniline. These are similar in molecular weight and composition to the isomerides of lower melting-point, and it is interesting to note that whereas dibenzylketone and benzal-*m*-nitraniline give a pure α-additive compound, deoxybenzoin with the same substance only gives the isomeride of higher melting-point.

DISCUSSION.

Dr. LOWRY asked if it was not possible that one of the three modifications described by the author as the α-, β-, and γ-forms might be a stable mixture of two isomerides and not a single chemical compound. In the case of the three modifications of π-bromonitrocamphor, those melting at 108° and 142° were found to be the normal and pseudo-nitro-compounds respectively, whilst that melting at 126° was only a stable mixture of these two.

Dr. FRANCIS, in reply, said that he had been unable to obtain any evidence that any of the modifications described was a mixture, and that if they were mixed they could be again separated by fractional crystallisation.

*34. "*Mesoxalic Semi-aldehyde.*" By H. J. H. FENTON, F.R.S., and J. H. RYFFEL, B.A., B.Sc.

Chlorine is slowly absorbed by a solution of tartaric acid containing iron in the ferrous state, and the product, after removal of the unaltered tartaric acid, is found to have powerful reducing and other aldehyde properties. With phenylhydrazine, it yields a bright orange precipitate having the composition $C_{15}H_{14}N_4O_2$, which is identical in every respect with the osazone obtained by Nastvogel from dibromo-pyruvic acid (*Annalen*, 1888, ccxlviii., 85) and by Will from collodion-wool (*Ber.*, 1891, xxiv., 400 and 3831).

A re-investigation, however, proves that the true melting-point of this osazone is 222—224° instead of the lower number 201—207°, ascribed to it by various authors. This osazone has the constitution $CH:N_2HPh\cdot C:N_2HPh\cdot CO_2H$, and corresponds either to hydroxy-pyruvic acid, tartronic semi-aldehyde, or mesoxalic semi-aldehyde.

With hydroxylamine, however, the *dioxime*, $CHNOH\cdot CNOH\cdot CO_2H$, or dioximidopropionic acid, is obtained identical with that prepared by Söderbaum from dibromopyruvic acid (*Ber.*, 1892, xxv., 904) and by oxidation with cupric hydroxide in alkaline solution *mesoxalic acid* results.

Since the oxidation product is free from chlorine, these facts leave no room for doubt as to the nature of the substance in question, namely, that it is the semi-aldehyde of mesoxalic acid.

Its formation from tartaric acid in the manner above described is due to the initial production of dihydroxymaleic acid, and it may be prepared in a pure state directly from the latter acid by the action of ferric salts. With ferric sulphate or chloride, for example, the change takes place almost quantitatively according to the equation $C_4H_4O_6+Fe_2(SO_4)_3=C_3H_2O_4+2FeSO_4+H_2SO_4+CO_2$.

After removal of the iron salt and free mineral acid, the product is obtained as a thick syrup, which, so far, has not been induced to crystallise. The derivatives and transformations of this aldehyde-acid are being further investigated.

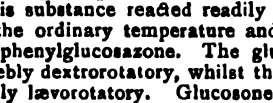
*35. "*The Action of Hydrogen Peroxide on Carbohydrates in the presence of Ferrous Salts.*" III. By R. S. Morrell and J. M. Crofts.

The specific action of hydrogen peroxide in the presence of ferrous sulphate was first demonstrated by Fenton in the oxidation of tartaric acid to dihydroxymaleic acid (*Trans.*, 1894, lxv., 899). Glucose, lævulose, arabinose, and rhamnose have been shown by the authors to be transformed by this peculiar reaction into osones, which were recognised by their power to react with substituted hydrazines at the ordinary temperature (*Trans.*, 1899, lxxv., 786; and 1900, lxxvii., 1219). It has been found that mannose on oxidation gave an osone which yielded with phenylhydrazine at the ordinary temperature phenylglucosazone. The preparation of pure glucosone from both glucose and levulose has been attempted, and a white amorphous solid has been obtained which gave analytical results agreeing with the formulæ $C_6H_{12}O_6$ and $C_6H_{10}O_6$. This substance reacted readily with phenylhydrazine at the ordinary temperature and furnished a good yield of phenylglucosazone. The glucosone from glucose was feebly dextrorotatory, whilst that from lævulose was slightly lævorotatory. Glucosone obtained by E. Fischer (*Ber.*, 1889, xxii., 87) is lævorotatory.

An aqueous solution of glucosone, prepared from either glucose or lævulose, on oxidation with bromine at 40° furnished a good yield of the calcium salt of a trioxybutyric acid. The barium salt of the same acid has been obtained, and both the calcium and barium salts seem to be identical with *d*-erythronic acid (Ruff, *Ber.*, 1899, xxxii., 3678). The calcium and barium salts of this acid obtained from glucosone prepared from either dextrose or lævulose have yielded butyric acid on reduction with hydriodic acid and phosphorus.

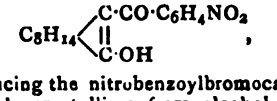
*36. "m-*Nitrobenzoylcamphor.*" By M. O. Forster and Miss F. M. G. Micklethwait.

α'-m-*Nitrobenzoyl-α-bromocamphor*,—



prepared from α'-benzoyl-α-bromocamphor and fuming nitric acid, separates from methyl alcohol in pale yellow flat prismatic needles melting at 93—94°; a 2 per cent solution in chloroform has $[\alpha]_D = +87\cdot9°$. α-m-*Nitrobenzoyl-α'-bromocamphor*, obtained in a similar manner from the isomeric benzoylbromocamphor, crystallises in aggregates of small pale yellow needles and melts at 101—102°; a 2 per cent solution in chloroform has $[\alpha]_D = -26\cdot1°$.

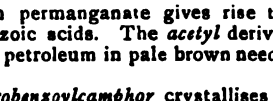
Enolic m-*nitrobenzoylcamphor*,—



formed on reducing the nitrobenzoylbromocamphors with alcoholic potash, crystallises from alcohol in long pink silky needles melting at 106—107°; it has not been obtained in the ketonic form, but the specific rotation of a 2 per cent solution in chloroform, having $[\alpha]_D = +209\cdot5°$ when freshly prepared, falls to $+200\cdot1°$ in the course of several days, indicating slight transformation into the isomeride. *m*-Nitrobenzoylcamphor dissolves in aqueous alkali hydroxides and the alcoholic solution develops an intense purple colouration with ferric chloride; oxidation with potassium permanganate gives rise to camphoric and *m*-nitrobenzoic acids. The *acetyl* derivative crystallises from light petroleum in pale brown needles and melts at 127—128°.

Enolic o-*nitrobenzoylcamphor* crystallises from alcohol in pale brown transparent prisms and melts at 118°; a 2 per cent solution in chloroform has $[\alpha]_D = +44\cdot5°$ when freshly prepared, and $+60\cdot5°$ after an interval of several days.

α'-m-*Nitrobenzoyl-α-chlorocamphor*,—



crystallises from alcohol in aggregates of pale yellow prisms and melts at 72—74°; a 2 per cent solution in chloroform has $[\alpha]_D = +40\cdot4°$.

α-m-*Nitrobenzoyl-α'-chlorocamphor* separates from alcohol in small nearly colourless needles and melts at 110°; a 2 per cent solution in chloroform has $[\alpha]_D = +7\cdot1°$. When these isomerides are heated with alcoholic potash, enolic *m*-nitrobenzoylcamphor is not formed; the sole products are α-chlorocamphor and *m*-nitrobenzoic acid.

*37. "*The Cloëz Reaction.*" By F. D. Chattaway and J. M. Wadmore.

The reaction between cyanogen chloride or bromide and an alcoholic solution of sodium ethylate appears at first sight to be inconsistent with the nitrogen halogen constitution which we have shown cyanogen chloride and bromide to possess. It becomes intelligible, however, when we consider the behaviour of the nitrogen halogen linkage towards alcohol.

Halogen attached to nitrogen reacts very readily with alcohol, the halogen being invariably replaced by hydrogen and a hypohalogen ester formed thus:—
$:N-X+C_2H_5OH=:N\cdot H+C_2H_5OX$.

The interaction of ethyl alcohol and trichloriminocyanuric acid, when ethyl hypochlorite and cyanuric acid are produced, is a simple case. In the Cloëz reaction, cyanogen chloride or bromide first reacts with the alcohol as a typical nitrogen halogen compound, forming hydrocyanic acid and ethyl hypochlorite or hypobromite, thus:—
$C:N\cdot Cl+C_2H_5OH=C:N\cdot H+C_2H_5OCl$.

These then combine and form ethyl iminochlorocarbonate, the hydrocyanic acid, or sodium cyanide, since sodium ethylate is present, behaving as an unsaturated substance:—

$$C_2H_5OCl+C:N\cdot H=\overset{C_2H_5O}{\underset{Cl}{}}>C:N\cdot H.$$

The ethyl iminochlorocarbonate then reacts in various ways to form the different substances which have been isolated from the product of the action; for example, it reacts with alcohol to form diethyl iminocarbonate, with water to form urethane, whilst three molecules interact with elimination of hydrogen chloride to produce normal triethyl cyanurate.

DISCUSSION.

Professor TILDEN thought Dr. Chattaway had brought forward a body of evidence which practically established his case, but he would like to inquire whether the production of a nitrile, by interaction between a common cyanide and a haloid compound, had engaged the attention of the authors, and what explanation they would give of the mechanism of the change. In such interactions, carbon appears to be directly attached to carbon, and there is no evidence of even a temporary formation of a compound in which the cyanide group is linked on by means of the nitrogen.

Dr. CHATTAWAY, in reply, stated that the imino-structure of cyanides admitted of action occurring in three ways—addition to the nitrogen, or to both nitrogen and carbon, or to carbon, taking place thus:— $H{\cdot}N{:}C+X_2=HX_2{:}N{:}C$; or $H{\cdot}N{:}C+X_2=H{\cdot}NX\,CX$; or $H{\cdot}N{:}C+X_2=H{\cdot}N{:}C{:}X_2$, the mode of interaction in any particular case and the further secondary changes being determined by the nature of the cyanogen derivative, and by that of the substance interacting with it. All reactions of these compounds could be formulated according to one or other of these types. The imino-formulæ of the cyanides are capable of expressing conventionally three phases of atomic relationship, the atoms composing a single molecule can only be in one phase at any instant, but the particular phase at which it reacts is determined by a related phase of the interacting molecule.

38. "*The Picrimidothiocarbonic Esters.*" By J. C. CROCKER, B.A.

When picryl chloride is treated with ammonium thiocyanate in alcoholic solution, a reaction readily takes place from which a well crystallised yellow substance is obtained, having the empirical formula $C_{15}H_9N_7SO_{13}$. On hydrolysis, it yields picramide. The reaction may be thus explained. Picryl thiocarbimide, $Pi{\cdot}N{:}C{:}S$, is probably the first product. A molecule of this substance adds on another molecule of picryl chloride, the chlorine atom becoming attached to the carbon atom and the picryl group to the sulphur atom. The acid chloride thus formed reacts with the alcohol to form a picrimidopicrylthiocarbonic ester with the elimination of hydrochloric acid. Thus:—

$$Pi{\cdot}N{:}C{:}S+Pi{\cdot}Cl \longrightarrow Pi{\cdot}N{:}C\!<\!{}^{Cl}_{S-Pi} \longrightarrow$$

$$\longrightarrow Pi{\cdot}N{:}C\!<\!{}^{Cl}_{S{\cdot}Pi} + EtOH \longrightarrow PiN{:}C\!<\!{}^{OEt}_{S{\cdot}Pi}.$$

Similar substances have also been prepared from methyl, propyl, and *iso*butyl alcohols. In the case of methyl alcohol, two substances have been obtained, which are stereoisomeric, corresponding to the "syn-" and "anti-" forms of the oximes—

$$CH_3{\cdot}O{\cdot}C{\cdot}S{\cdot}Pi \quad \text{and} \quad CH_3{\cdot}O{\cdot}C{\cdot}S{\cdot}Pi$$
$$\quad\quad\quad

prepared from tetrabromoaniline, silky, white needles melting at 228—229°; 2:3:4:6 *tetrabromodiacetanilide*, $C_6HBr_4 \cdot NAc_2$, lustrous, four-sided prisms with domed ends, melting at 164°. 2:3:6 *Tribromo 4-nitroaniline*, $C_6HBr_3(NO_2) \cdot NH_2$, prepared by brominating 3-bromo-4-nitroaniline, pale lemon-yellow needles, melting at 155—155·5°. 2:3:4-*Tribromo-6-nitroaniline*, $C_6HBr_3(NO_2) \cdot NH_2$, prepared from 3-bromo-6 nitroaniline, orange-yellow needles, melting at 165·5—166°; 2:3:4-*tribromo-6-nitroacetanilide*, $C_6HBr_3(NO_2) \cdot NHAc$, colourless, flattened needles melting at 221°.

42. "*The Resolution of Pheno-α-aminoheptamethylene into its Optical Isomerides. Tartrates of Pheno-α aminoheptamethylene and of Hydrindamine.*" By F. S. KIPPING and A. E. HUNTER.

An investigation of the salts produced by the combination of *dl* pheno-α-aminoheptamethylene with tartaric acid has shown that this base behaves quite differently from *dl*-hydrindamine (pheno α-aminopentamethylene) in spite of the similarity in constitution of the two bases.

dl-Pheno α-aminoheptamethylene-d-tartrate,—

$$2C_{11}H_{15}N,C_4H_6O_6+\tfrac{1}{2}H_2O,$$

separates from neutral and from acid solutions of the *dl*-base in aqueous tartaric acid, in lustrous prisms decomposing at about 235°; in aqueous solution $[\alpha]_D = +13°$.

l-Pheno-α-aminoheptamethylene hydrogen d-*tartrate*, $C_{11}H_{15}N,C_4H_6O_6+3H_2O$, is obtained when the solution of the normal tartrate is mixed with excess of tartaric acid and fractionally crystallised; it forms slender prisms, and melts at 181—182°; its aqueous solutions are feebly lævorotatory ($[\alpha]_D = -1·6°$ approximately).

l-Pheno-α-aminoheptamethylene d-*tartrate*, $2C_{11}H_{15}N,C_4H_6O_6$, prepared from the *l* base, crystallises in transparent, triangular plates, and decomposes at 215—217°. The *hydrochloride* of the *l*-base forms lustrous prisms, and in dilute aqueous solution its specific rotation is $[\alpha]_D = +24°$.

Benzoyl-l-pheno-α-aminoheptamethylene, $C_{11}H_{14}N \cdot COPh$, crystallises in needles, melts at 175—176°, and is dextrorotatory in methyl alcoholic solution.

d-*Pheno-α-aminoheptamethylene* d-*tartrate*,—

$$2C_{11}H_{15}N,C_4H_6O_6,$$

can be isolated from the mother-liquors obtained after separating the hydrogen salt of the *l*-base; it is anhydrous and decomposes at about 216—217°.

d-*Pheno-α-aminoheptamethylene hydrogen tartrate*, $C_{11}H_{15}N,C_4H_6O_6$, prepared by treating the normal salt with tartaric acid, decomposes at about 205—206°.

dl-Hydrindamine hydrogen d-*tartrate*,—

$$C_9H_{11}N,C_4H_6O_6+H_2O,$$

crystallises in slender needles, melts at 168—169°, and is not resolved or changed in any way by repeated crystallisation from water. The normal d-*tartrate* of the *dl*-base forms plates melting at about 200°, and dissociates hydrolytically in aqueous solution.

l-Pheno-α-aminoheptamethylene d-*bromocamphorsulphonate*, $C_{11}H_{15}N,C_{10}H_{14}BrO \cdot SO_3H$, crystallises in prisms melting at 216—217°; in dilute aqueous solution $[\alpha]_D = +47·8°$.

PHYSICAL SOCIETY.

Ordinary Meeting, March 14th, 1902.

Mr. S. LUPTON, Vice-President, in the Chair.

A PAPER on "*The Thermal Expansion of Porcelain*" was read by Mr. A. E. TUTTON.

The paper gives an account of experiments made to determine the expansion of Bayeux porcelain between 0° and 180° C. The material employed was a portion of the tube used by Bedford in his experiments on the expansion of porcelain between 0° and 830° C. Another piece of the same tube has also been used by Chappuis in a series of determinations by the Fizeau method between 0° and 83° C. The author has worked with an interference dilatometer, which possesses advantages over the Abbe form of the original Fizeau apparatus. The observing part of the instrument is separated from the expansion chamber, and the temperature of the interference tripod and the substance under investigation, which it carries, is measured by means of a thermometer bent just above the cylindrical bulb, and so arranged that the latter lies on the tripod table. The chief advantages over the Fizeau apparatus are briefly:—1. The employment of a micrometric method of measuring the position and width of the interference bands. 2. The use of autocollimation. 3. The employment of C hydrogen light. 4. An arrangement of the thermal chamber which readily permits an extension of the range to 120° C. The author has also introduced an aluminium compensator, a relatively thick disc of aluminium laid on the top of the porcelain tube. This overcomes the difficulty of polishing the porcelain, and affords a large field of bands instead of an annular ring showing parts of bands. The mean of three determinations with three specimens of material gives the following result for the linear expansion $L_t = L_0[1+10^{-6}(2522t+7·43t^2)]$. The results presented by the author agree tolerably well with those of Chappuis, but the constant *a* is slightly smaller. The constant *b* is seven times larger than according to Bedford. The discrepancies between the results of Chappuis and Bedford appear to be due to a fundamental real difference, dependent on the interval of temperature for which the determinations were made. The increment per degree of the coefficient of expansion of porcelain is not a constant quantity, but one which is much larger between 0° and 100° than at higher temperatures. The supposition of Prof. Callendar, as to the anomalous expansion between 0° and 100°, appears to be well founded.

The SECRETARY read a letter from Dr. P. E. SHAW stating that the accuracy of the micrometer readings seemed to depend more on the distinctness of the rings than on anything else. He asked if the author considered mercury or hydrogen light better than sodium light, and if the error in the present research was less than $\frac{\lambda}{25}$, which is supposed to represent the accuracy of the Abbe-Fizeau method.

Dr. CHREE expressed his interest in the paper because of its bearing on the measurement of temperature by the gas thermometer, in which case it is essential to know the coefficient of expansion of the porcelain bulb. He did not consider the behaviour of porcelain anomalous, because an irregular expansion might be expected. In the case of Jena glasses used in thermometry, the values of *a* and *b* determined between 0° and 100° are different to those obtained when the range is extended to 200° C. He was surprised at the differences between Bedford and Chappuis and Tutton, and said that the evidence was in favour of the instruments used by the latter observers. It would be interesting if the author could obtain Chappuis's specimen of porcelain, and repeat his experiments between 0° and 80°.

Mr. TUTTON, in reply to Dr. SHAW, said that the bands were perfectly distinct when using red hydrogen light. He did not use mercury light because of the necessity of constantly heating the tube.

The SECRETARY then read a paper by Mr. W. WILLIAMS on "*The Temperature Variation of the Electrical Resistances of Pure Metals and Allied Matters.*"

In the first part of the paper an attempt is made to correlate the periodic variations which pure metals exhibit as regards their atomic weights, chemical valencies, melting-points, and electric resistances. If *m* is the chemical valency, V the atomic volume, θ the absolute temperature, T the absolute melting-point, and *c*, or $V^{\frac{1}{3}} \propto T$ the constant of Pictet's law, then $\epsilon \propto \frac{mV\theta}{cT}$, where

σ is the specific resistance at 0° C. This relation holds for most of the metals, but fails for gold, indium, tin, and aluminium, and also for metals of the iron group. The temperature resistance-coefficients of pure metals are not equal to $\frac{1}{273}$, and an expression for the change of resistance with temperature has been deduced which holds approximately for many metals. The author also obtains simple expressions for the average increment per degree of the specific heat of metals, and for the ratio between the specific resistances of the solid and liquid states of a metal at the temperature of fusion.

A paper entitled "*A Suspected Case of Electrical Resonance of Minute Metal Particles for Light Waves. A New Type of Absorption*," by Prof. R. W. WOOD, was read by the SECRETARY.

Experiments on which the author has been engaged have led him to believe that he has found a new type of light absorption, which it may be possible to refer to the electrical resonance of small metallic particles for waves of light. Metallic deposits on glass have been produced which are shown by the microscope to consist of particles less than the wave-length of light, and which by transmitted light exhibit colours as brilliant as those produced by aniline dyes. The author has sought to explain these colours by interference and diffraction, and has been forced to accept the hypothesis suggested in the title of the paper. The metallic deposits can be obtained by heating small fragments of the alkali metals in exhausted glass bulbs, when the vapour condenses on the cold parts of the bulbs and forms the films. It can be shown that the colours are due to the presence of metallic sodium (in the case in which sodium has been used) by allowing air to enter the bulb; oxidation takes place, and the film vanishes. In some experiments the air has been allowed to enter very slowly, and the changes which the film undergoes before it vanishes have been examined. The particles which form the deposits can be classed under three heads; (1) coarse particles which diffract or scatter light and give the bulb a silky lustre; (2) minute particles very close together which regularly reflect those wave-lengths absent in the transmitted light, but give no scattered light; and (3) minute particles far apart which diffuse light of the same wave-lengths as those which are to some extent absent from the transmitted light. By observing the spectrum of the transmitted light the author has examined the changes in colour which accompany changes in temperature of the films. The paper gives an account of the relation between the colour of the film and the size and distribution of the particles, and also of the behaviour of the films with polarised light. Experiments upon the electric resistance of the films have proved that they are non-conducting. The author concludes by stating that at the present stage it is impossible to decide either in favour of, or against, the theory of resonance. The idea of resonance has proved a useful working hypothesis for explaining some of the phenomena described in the paper.

The SECRETARY read a letter from Prof. R. THRELFALL drawing the attention of the author to some experiments upon the same subject published by him in 1894. Particles of gold and platinum were deposited in a liquid, and good scattering of light obtained in all cases. The polarisation angle was exactly the same as with non-conducting particles. Prof. J. J. Thomson has shown that the peculiar scattering effect is only to be expected within very narrow limits as to size of particles. The colours observed by Prof. Wood should only be noticed when the particles have a very restricted size, and evidence in favour of or against the author's theory should be afforded by comparing the observed and calculated sizes of the particles. The value of the polarisation angle of the scattered light observed by Prof. Wood is evidence in favour of the theory suggested.

The CHAIRMAN pointed out that the experiments had probably been rendered difficult by the different colours in which the oxides of sodium and potassium can exist.

The Society then adjourned until April 11th.

NOTICES OF BOOKS.

Inorganic Evolution as Studied by Spectrum Analysis. By Sir NORMAN LOCKYER, K.C.B., F.R.S., Professor of Astronomical Physics in the Royal College of Science, London, &c. London: Macmillan and Co. 1900. Pp. 198.

THE volume now before us contains an account of some of the author's recent inquiries into the chemistry of the stars, and of some questions which have grown out of these inquiries. He first describes the principles, methods, and application of the inquiry to the sun and stars. One of the first questions that arises is this:—Do the chemical elements make themselves visible indiscriminately in all the celestial bodies, so that practically, from a chemical point of view, the bodies appear to us of similar chemical composition? The answer to this question is in the negative.

From the spectra of those stars which resemble the sun, it is to be gathered that the atmospheres of some stars are chiefly gaseous, consisting of elements we recognise as gases here; others are chiefly metallic, while others, again, are mainly composed of carbon or of compounds of carbon. Three of the main groups of stars may be classified as follows:—

Gaseous stars	Longest spectrum
Metallic stars	Medium spectrum
Carbon stars	Shortest spectrum

and by associating the above with other observations it is reasonable to make the following general statement:—

Gaseous stars	Highest temperature
Metallic stars	Medium temperature
Carbon stars	Lowest temperature

Hence the difference in apparent chemical constitutions are associated with differences of temperature. Laboratory work has enabled the author to bring still a third class of facts into the question, by the help of the clèveite gas-spectrum and the spectrum of enhanced metallic lines, and the results are quite in harmony with the preceding ones:—

Gaseous stars ..	Highest temperature ..	Strong clèveite gas, and faint enhanced lines.
Metallic stars ..	Medium temperature ..	Feeble clèveite gas, and strong enhanced lines. No clèveite gas, and strong arc lines.
Carbon stars ..	Lowest temperature ..	Faint arc lines.

The chemical law, therefore, is this—that in the hottest stars we find, generally speaking, hydrogen, helium, asterium, and doubtless other still unknown elements, almost exclusively. At the next lowest temperatures these gases are being replaced by metals in the state in which they are observed in the laboratory when the most powerful jar-spark is employed. At a lower temperature still, the gases almost disappear entirely, and the metals exist in the state produced by the electric arc.

The author then passes from general to particular results, and gives in detail the results of his observations on stars as hot as, or hotter than Arcturus, this star being taken as representing the temperature of the sun.

The chemical classification of stars is the next point dealt with, but it must be understood that only the most predominant chemical features are considered, and that there is no sharp line of separation between the larger groups.

In 1873 the author suggested that many difficulties would vanish if it were conceded that the "atoms" of the chemist were broken up or dissociated by the high temperatures employed in the new method of investigation.

Some of the views brought forward from time to time have received general acceptance, but the author's view as to the subsequent dissociation of molecules, when once the line spectrum stage has been reached, was still rejected by many. The various points involved are then dealt with seriatim, under the headings of Stellar evidence, "Series" evidence, "Shifting" of lines evidence, Magnetic pertubation evidence, and Fractionation evidence.

From the above it is reasonable to suppose that the spectrum of an element is produced not by similar but by dissimilar molecules.

The next phase of the subject considered by the author is the objections raised to the dissociation hypothesis. He has supposed, and he thinks legitimately until the contrary is proved, that the materials out of which, on the meteoritic hypothesis, worlds are eventually formed are similar in all parts of space, and he contends from stellar evidence that the facts are distinctly against the view of "different chemical parishes in space." The Sun, Arcturus, and Capella, and other cooling stars, enormously separated in space, contain the same spectral lines with almost identical intensities, so that not only do they contain the same elements, but they contain them "in absolutely identical proportion." We are therefore justified in the conclusion that the differences recorded in stellar spectra do not come from a different percentage composition of the elements present, but arise from the action of different temperatures on the same molecules.

The next important point is the discussion as to the distribution of the various chemical groups of stars, not only in their relation to their direction in space as seen from the solar system, but also in relation to their distance from us, and the results arrived at are summarised in Chapter XVII.

It is found that the gaseous stars are chiefly in the Milky Way, and are far away from us; the proto-metallic stars are not so confined to the Milky Way, and they are not so far away from us; but the metallic and carbon stars have not much connection with the Milky Way, and they are close to us. Finally, the author is led to the conclusion that there is no localisation of the chemical elements so far as *direction* is concerned.

The final chapters of this book deal specially with inorganic evolution, and it is argued that as evolution depends upon time in organic, so does it depend upon temperature in inorganic nature.

It is well known that the effect of high temperatures is to produce simplifications of matter; dissociation in all its stages must reveal to us the forms, the coming together of which has produced the thing dissociated or broken up by heat. If this be so, the final products of dissociation must be the earliest chemical form. The earlier chapters of this book answer the question fully; it is shown how in cosmical evolution we deal with a continuity of effects accompanied by considerable changes of temperature, from the gradual coming together of meteoric swarms until eventually we have a mass of matter cold and dark in space. The various stars which represent these changes have been arranged along a so-called temperature curve. On one branch the stars get hotter until at the top we find the hottest stars that are known, then on the descending branch we have the cooling bodies coming down in temperature until we reach that of a dark world, like the companion of Sirius, or our own earth.

The Colouring Matter Industry ("L'Industrie des Matières Colorantes"). By Justin Dupont, Professor at the "Institut Commercial." With 34 Figures in the Text. Paris: J. B. Baillière and Sons. 1902. Pp. 360.

The colouring matter industry is one of great importance, not only on account of the enormous development of the allied industries, but also because of the scientific interest which surrounds these products, and the theoretical discussions which are carried on with regard to their constitution. In fact the study of colouring matters now forms a definite branch of chemical science, and the author hopes that this elementary volume on this branch of chemistry will be found useful to those who are studying the subject.

The plan adopted in arranging this book is both simple and logical; after a short review of the general theory of colouring matters, he enters on the subject itself, and divides its treatment into three parts, viz., natural colouring materials, artificial colouring materials, and the application of colouring materials.

The first part gives a full and interesting account of the history, properties, and characteristics of natural dyes.

In part two (which forms the bulk of the volume) the author first deals with the matters extracted from coal-tar, then the intermediary derivatives between them and the actual colouring matters. These chapters contain a great deal of useful information as to the constitution and preparation of many of these intermediary products. He then deals with the colouring matters themselves, and he has adopted the classic divisions by which the colouring materials are divided into a certain number of groups or families, according to their chemical functions. He fully describes the characteristics of each of these series of bodies, their manner of formation, and their properties.

The third part of the volume describes the methods use for the application of colouring matters, and the properties of the principal fibres in connection with which they are used, such as silk, wool, cotton, &c. The wide differences in the properties of these different fibres necessitates the use of special methods for each; these are described in a clear and lucid manner.

The book is furnished with a full table of contents and a good index.

Practical Chemistry. By R. Abegg and W. Herz. Translated by H. T. Calvert, B.Sc. (Vict.). London: Macmillan and Co. New York: The Macmillan Company. 1901.

The scope and object of this book are indicated by the secondary title "An Experimental Introduction to Laboratory Practice and Qualitative Analysis from a Physico-chemical Standpoint." Such a work as this is a complete novelty in the English language, and the introduction of a considerable amount of theoretical discussion in a practical book is regarded as somewhat unusual, though in our opinion by no means to be deprecated. The early introduction of the theory of ionisation tends to simplify many problems of analytical chemistry, and above all does much to prevent the subject becoming a mere test-tubing exercise, though a discussion of the theory in its fullest developments would make too great demands on the student's knowledge. Such books as these, in which the causes of reactions are more emphasised than the visible effects, possess a very distinct value of their own.

The book opens with a short course of preliminary preparations of the most common inorganic substances, followed by a chapter on the theoretical foundations of analysis. Here such subjects as the law of mass action are dealt with and dismissed in a few pages, but as far as they go the accounts appear satisfactory summaries. The section on the reactions of the kations and anions exhibits no great difference from those of the ordinary type of analytical hand-book, except for the continual references to, and explanations by means of, the ionisation theory; in some places the introduction of more formulæ might be advocated, as, for example, in the case of the thio-acids of tin, &c.

The system adopted for the detection and qualitative separation of the elements is based chiefly upon Wallach's analytical tables, but the authors claim that the method of separating the acids is new. This part of the book is particularly well arranged, and the tables of the atomic weights of the elements and the periodic system give the results of the most recent determinations.

Leçons sur la Théorie des Gaz. Première Partie. By L. BOLTZMANN. Translated by A. GALLOTTI. Paris: Gauthier-Villars. 1902.

THESE lessons on the theory of gases are provided with an introduction and notes by M. Brillouin, of the Collège de France. The introduction shortly reviews the historical development of the molecular hypothesis in the theory of gases, and forms a suitable preface to the detailed study of the subject. This volume deals with the molecules considered firstly as elastic spheres, and, secondly, as centres of force, the third chapter being occupied with the investigation of the repulsive force varying inversely as the fifth power of the distance. The law of distribution of velocities, and the mathematical signification of Boltzmann's magnitude H, call forth some valuable criticisms by M. Brillouin, who is not altogether satisfied with some of Boltzmann's conclusions.

This work is adapted for those students of physics, who, having already a fair knowledge of the theory of gases, require a book in which recent work on the subject is collected in convenient form, and attempts made to do something towards the elucidation of some of the many difficulties. Though occasionally the author appears to us somewhat too daring, he is never obscure, and every mathematical step is carefully and precisely worked out.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees o temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxiv., No. 8, February 24, 1902.

Properties of Azobenzene and Hydrazobenzene.—F. Freundler and L. Béranger.—The authors apply Friedel and Craft's reaction in the attempt to prepare azoic compounds and their derivatives. They find that this reaction cannot be employed for the preparation of the azoic compounds with ketonic function, and so employ other methods. The diacetylhydrazobenzene can be prepared by heating hydrazobenzene for several hours to boiling point, with excess of acetic anhydride. By using a commercial product very rich in azobenzene they obtain, instead of a diacetyl derivative, a molecular combination of this latter with azobenzene. This substance crystallises without dissociating in large orange-coloured prisms, melting at 98·5—99°. It corresponds to the formula $2[C_6H_5.N(COCH)_3].N.(COCH_3).C_6H_5.N=N.C_6H_5$.

Constitution of Dibutylic and Dicœnanthylic Alcohols.—Marcel Guerbet.—The author has already shown that dibutylic alcohol, $C_8H_{18}O$, can be prepared by heating normal butylic alcohol with its sodium derivative to about 200°. A series of experiments on this substance lead him to attribute to dibutyric acid the formula—

$$CH_3—CH(CH_2—CH_2—CH_2—CH_3)—CO_2H$$

and to dibutylic and dicœnanthylic alcohols respectively the constitutional formulæ—

$$CH_3—CH(C_4H_9)—CH_2—CH_2OH$$

$$CH_3—(CH_2)_3—CH(C_7H_{15})—CH_2—CO_2H$$

The Polymerised State of Ordinary Indigotine and the Isomeric Transformation of Indigotine into Indirubine.—L. Maillard.—The blue of indigo, when extracted by chloroform, cannot be obtained in a crystalline form, and thus differs from indigotine in at least two properties—(1) its much greater solubility in chloroform; (2) the ease with which it is transformed into indirubine by means of acid chloroform. These differences are explained if the two blue colouring matters are considered as polymers the one of the other, and by attributing naturally the most complicated formula to the most stable.

Certain Reactions obtained by means of Magnesium Amalgam.—L. Meunier.—Magnesium amalgam, even in the cold, attacks absolute ethylic alcohol, hydrogen being evolved and the corresponding ethylate separating out. This ethylate, when washed and rapidly dried, is in the form of a white powder, corresponding to the formula—

$$Mg<^{OC_2H_5}_{OC_2H_5}.$$

Magnesium amalgam can also be used for the preparation of the saturated carbides, as it reduces the alcoyl iodides in presence of absolute alcohol. Ethylic aldehyde acts very violently on the amalgam, but the reaction can be moderated by adding anhydrous benzine aldehyde or a sufficient quantity of anhydrous ether. A syrupy liquid is obtained which contains a magnesium compound in solution.

Constitution of Tariric Acid.—M. Arnaud.—Tariric acid is the principal constituent of the glyceride which can be extracted from a certain grain. The author has shown that this acid is an isomer of stearolic acid, and that it can be integrally transformed into stearic acid by the action of hydriodic acid and red phosphorus at 210°. This acid, thus prepared, has a formula $C_{18}H_{32}O_2$, and belongs to the series of non-saturated acids, $C_nH_{2n-4}O_2$. It ought to contain a triple acetylenic liaison like stearolic acid. This latter fact the author proves by a series of experiments on the acid and its salts, and proposes to examine the products of oxidation of the acid.

New Method of Identifying the Pseudo Acids. Application to the Oximido-cyanacetic Ethers.—P. T. Muller.—The oximido cyanacetic ethers are identified by the joint presence of the isonitroso-radicle =NOH and the negative radicle —CN, which give to the compounds decided acid properties. The electric conductivity of their aquous solutions also place these bodies near to acetic acid. They can be accurately titrated against phenolphthalein, and their soda-salts do not show appreciable hydrolysis. Their aqueous solutions are colourless, with a slight yellow tinge, the alkaline salts being yellow both in the solid state and in solution. However, their quarter normal solutions are transparent enough for the refractive power to be measured. A table is given of the optical measurements.

Some Derivatives of Methyl-nonyl-ketone. — H. Carette.—Continuing his research on the derivatives of methyl-nonyl-ketone, the author transforms this acetone into nitrile-alcohol by fixation of hydrocyanic acid. By immediate hydration of this nitrile-alcohol he obtains the ordinary corresponding acid-alcohol, and in addition an amido-alcohol resulting from the fixation of a single molecule of water on to the alcohol nitrile.

Researches on the Fatty Acids present in Contaminated Waters.—H. Causse.—The presence of fatty acids themselves in water is not injurious, but as a rule it indicates contamination. When their proportion is about 0·01 m.grm. per litre their influence on the water is slight but perceptible. They discolour sulphurous violet slowly and incompletely. Para-diazobenzene-sulphonate of sodium gives a slight yellow colouration, without a trace of orange. If, however, the quantity of fatty acids increases, the oxygen in solution is reduced, and the reactions for pure waters disappear, giving place to those of contaminated waters.

MISCELLANEOUS.

The Variations of Temperature Accompanying the Solidification of Fused Organic Bodies.—Br. Pawlewski.—The author fuses a few cubic centimetres of an organic substance in a test-tube, and plunges a thermometer therein; then, while allowing the substance to cool rather slowly, he observes the thermometer every twenty seconds when near the point of solidification, and plots the figures on a curve in functions of time. According to the character of the curves, he is able to separate the substances examined into three classes. In the first type the ordinate sinks rapidly below the normal point of solidification *s* (that observed in a capillary tube), then remains constant; it then sinks again so that the two arcs of the curve separated by the median line show opposing concavities. The second type only differs from the first in the suppression of the median line, and gives a curve with no inflection for a temperature below *s*. In the third type the temperature, after falling considerably below *s*, rises again very rapidly, and remains constant for some time at a little below *s*; the rest of the curve is like that of the first type. Agitation causes substances of the third type to behave like those of the first, by destroying the surfusion. The falls below *s* are sometimes considerable (41° in the case of acetyldiphenylamine). Isomerous bodies often belong to different types.—*Berichte*, vol. xxxiii., p. 3727.

The Separation of Arsenic.—M. Rohmer.—The method consists in the distillation of the arsenical liquor in a current of hydrochloric and sulphurous acid gases (the latter is to reduce the As_2O_5); the arsenic is then given off as $AsCl_3$, and is collected in a cooled receptacle containing water. Bunsen observed (*Lieb. Ann. Ch.*, vol. cxcii., p. 321) that the reduction of the As_2O_5 is never complete, but the author, when operating in the presence of a little hydrobromic acid, obtained very satisfactory results. The operation is performed in a 500 c.c. flask; the solution to be examined is poured in with an excess of fuming hydrochloric acid, so as to fill one-third of the flask, and 1·5 grms. of potassic bromide or 1 grm. of bromine is added. It is then distilled for about fifteen minutes, while the current of hydrochloric and sulphurous acid gases are being passed through; the solution is thus reduced to about 40 c.c. The fumes are condensed first in a long tube, and then collected in a one-litre flask cooled down to 0°, and containing 300 c.c. of water; finally, the rinsings of the long tube are added to the contents of the flask. The arsenic can be estimated either gravimetrically in the form of As_2S_3, or volumetrically by means of a titrated solution of iodine. If either antimony or tin were present they would remain in the original flask.—*Berichte*, vol. xxxiv., p. 33.

Action of Zinc Powder on Bromide of Trimethylene—A. Volkof and B. Menschoutkine.—In a recent research the authors have shown that the action of zinc powder on bromide of trimethylene in alcoholic solution gives trimethylene mixed with propylene. If the alcohol is replaced by water, the reaction takes place at a temperature of about 70°, the disengagement of gas is rapid and regular, and the gas evolved is free from propylene, but even this gas is not pure trimethylene, it contains propane. It is impossible to describe the large number of experiments carried out to completely determine all the primary and secondary reactions which take place when zinc powder is made to react on bromide of trimethylene and its homologues under varying conditions; the conclusions to be drawn from the results only can be given:—1. The action of zinc powder and alcohol on bromide of trimethylene is analogous to that of these substances on the bromides of tetramethylene and pentamethylene; it produces a gaseous mixture formed of trimethylene, propylene, propane, and apparently a little hydrogen. 2. The presence of the propylene results from the formation, by reason of the action of the zinc oxide contained in the zinc powder, of an ether, $CH_2Br—CH_2—CH_2—O—C_2H_5$, which, with zinc and alcohol, gives propylene. 3. The propane results from the action of the zinc powder on the bromide of propyl produced by the incomplete reduction of the bromide of trimethylene. 4. The action of bromine on the gas obtained in the reaction of the zinc powder on the alcoholic solution of bromide of trimethylene gives the following products:—Bromides of propyl and of isopropyl coming from the propane, and being almost entirely transformed by the bromine into bromide of propyl, bromides of propylene, and of trimethylene, resulting from the propylene and from the trimethylene. 5. The speeds of the reactions of bromine with propane, propylene, and trimethylene are very different; it is for this reason that the whole of the propylene cannot be removed by means of bromine, but we cannot separate the trimethylene and the propane by this means; by the action of bromine on this mixture we obtain bromides of propylene and of trimethylene; the amount of the former is so slight as to escape notice. It may be added that up to the present pure trimethylene has never been obtained; with zinc and alcohol, a mixture of trimethylene, propylene, and propane is obtained; with zinc and water, trimethylene and propane; and with sodium, trimethylene and propane.—*Journ. Soc. Phys. Chim. R.*, vol. xxxii., p. 118.

MEETINGS FOR THE WEEK.

TUESDAY, 25th.—Society of Arts, 4.30. "The Sphere of State Activity in Australia," by the Hon. Sir John Alexander Cockburn, K.C.M.G.

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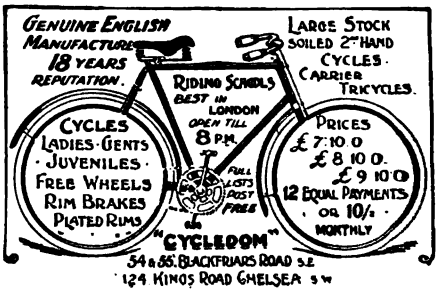
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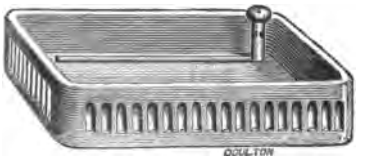

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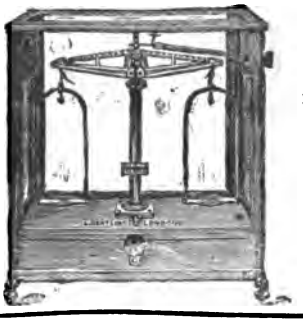

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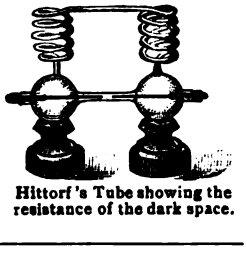

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THE CHEMICAL NEWS.

VOL. LXXXV., No. 2209.

THE DENSITY AND COEFFICIENT OF CUBICAL EXPANSION OF ICE.*

By J. H. VINCENT, D.Sc., B.A., St. John's College, Cambridge.

After an account of the methods employed by previous experimenters in the subject, reference is made to the views of Nichols, according to which two distinct kinds of ice have been subjected to experiments. The density of artificial ice is about 0·916 grm. per c.c., while that of natural ice is more than one part in a thousand greater.

Two tables are given showing in the first all the results which have been published concerning the density and coefficient of cubical expansion of ice, and in the second the same results tabulated separately according to the variety of ice used. The mean result for the density of natural ice at freezing-point is 0·9176, while that of artificial ice is 0·9162 grm. per c.c. Only one estimation of the dilatation of natural ice is available. It is 0·00011125 for the cubical coefficient per degree C. The mean of three available results for artificial ice is 0·000160.

It was thought desirable to use a method of experiment which would yield a result both for the density and for the coefficient of cubical expansion, and in order that the work should have any value it was necessary to employ some device other than any which had been used previously.

The method consisted in weighing a quantity of water in mercury. The water was weighed both as liquid at 0° C, and as solid at several temperatures below freezing-point. If we assume values for the density of water and mercury at 0° C., the density of ice at 0° C. can then be calculated, if we also assume that the densities of ice and mercury are linear functions of the temperature. The coefficient of cubical expansion of ice can also be calculated from these results, but it will depend on the law assumed for the contraction of mercury, and upon the accuracy of the thermometry.

Instead of using a sinker to keep the vessel containing the water or ice under the surface of the mercury, a modification of Joly's hydrostatic balance was employed (Joly, *Phil. Mag.*, September, 1888).

Ten values of the density of ice at different temperatures below 0° C. were obtained in this way. The specimens of water were four in number, and the temperatures ranged from −10·02° to −0·37° C. The whole of the weighings taken with the final form of apparatus are included in these determinations.

In one experiment the values obtained show unmistakably that the same specimen of water may assume different densities on freezing.

The ten values of the density are set out as functions of the temperature on a chart, and a graphical method is used to extrapolate for five values of the density of 0° C. These five values have weights assigned to them proportional to the number of separate determinations of the density from which they are derived. The numbers thus obtained and the weights to be assigned to them are set out in the table below.

Experiment.	Density at 0° C.	Weight assigned.
1	0·916335	3
2	0·915460	2
3	0·916180	2
4	0·915540	1
	0·916060	2
Weighted mean	0·9160	

We thus obtain 0·9160 grm. per c.c. as the density of ice at 0° C.

Four values for the coefficient of cubical expansion can be obtained from these results. They are:—

Experiment.	Coefficient of cubical expansion.
1	0·000155
2	0·000152
3	0·000153
4	0·000148
Mean	0·000152

The results of this investigation are that Nichols's value for the density of artificial ice is confirmed, but that since the same specimen of water can freeze into specimens of ice having different density, the use of the Bunsen ice calorimeter in absolute determinations must be limited to an accuracy of probably one in a thousand.

The expenses of this research have been in part defrayed by a Goverment grant from the Royal Society, and in part by the Cavendish Laboratory.

I wish to thank Professor J. J. Thomson, F.R.S., for his kind encouragement, and my thanks are also due to Mr. E. H. Griffiths, F.R.S., through whom I was led to undertake the investigation.

* Abstract of a Paper read before the Royal Society, Feb. 6, 1892.

ON CERTAIN CHEMICAL AND PHYSICAL PROPERTIES OF HÆMOGLOBIN.*

By ARTHUR GAMGEE, M.D., F.R.S., Professor Emeritus of Physiology in the Owens College.

This lecture consists of two parts, of which the first is bibliographical and critical, the second experimental.

Part I.—*Bibliographical and Critical.*

The author commences by stating that a peculiar interest—the parallel of that which in the plant organism belongs to chlorophyll—attaches to hæmoglobin, for, unlike any other chemical component of the animal body, in virtue of its special chemical and physical attributes, this remarkable substance may in the strictest sense be said to possess a definite and unique physiological function.

The author then discusses certain facts in reference to hæmoglobin and its products of decomposition which have a close bearing on his researches, or which possess special interest in the light of work which has been recently done. He does so under the following heads:—(1). The question of the identity of hæmoglobin throughout the animal kingdom. (2). The nature of the albuminous body which is linked to the coloured iron-containing group in hæmoglobin—*Globin* (*Hæmato-Histon*). Under this heading he discusses the general properties of the histons and their relation to the simplest of all the albuminous bodies, the *Protamines*. (3). The products of the decomposition of the iron-containing molecular group in hæmoglobin. Under this heading the author directs special attention to the researches of Schunck and Marchlewski, which have shown by the oxidation of phyllotaonin, one of the decomposition products of chlorophyll, a substance, PHYLLOPORPHYRIN, is obtained which is probably identical with hæmatoporphyrin. Under this, as also under the preceding head, the author discusses various questions in reference to the seat of the synthesis of hæmoglobin.

Part II.—*Experimental.*

(1). *Extension of Previous Observations on the Absorption of the Ultra-violet Rays of the Solar Spectrum by Hæmoglobin.*

The author refers briefly (illustrating his remarks by demonstrations) to his researches, previously published,

* Abstract of the Croonian Lecture read before the Royal Society, March 13, 1902.

into the absorption of the extreme violet and ultra-violet rays of the solar spectrum by hæmoglobin, its compounds, and certain of its derivatives. The region of the solar spectrum which he formerly investigated was that comprised between the lines F and Q (4861—3280).

He now examines the question whether oxy-hæmoglobin presents definite absorption for light of shorter wavelengths. Soret, whose observations were not conducted with solutions of hæmoglobin, but merely with diluted blood, observing by the aid of his fluorescent eye-piece the cadmium spark spectrum, found that diluted blood, in addition to the absorption band in the extreme violet, exhibited two additional bands. One of these, coinciding with the 12th cadmium line (3247), he considered to be probably due to hæmoglobin; the other, coinciding with the 17th cadmium line (2743), he assumed to be caused by serum albumin, his observations having previously shown that all albuminous and albumenoid bodies, with the exception of gelatin, are characterised by an absorption band in the position of the 17th cadmium line.

Employing solutions of many times crystallised oxy-hæmoglobin of great purity and of varying concentration, and with the aid of the sparks of a powerful induction coil, the author has obtained a series of photographs of the cadmium spark spectrum with and without the interposition of the solutions. The examination of these photographs shows that solutions of oxy-hæmoglobin which are sufficiently transparent to allow the ultra-violet spectrum of cadmium to be photographed, present no absorption bands corresponding either to the 14th or the 17th cadmium lines. The absorption band observed by Soret in correspondence with line 14 is therefore not due to the blood colouring matter, but to some other organic constituent present in the blood.

(2). *Behaviour of Oxy-hæmoglobin and* CO-*hæmoglobin in the Magnetic Field. The Intense Ferro-magnetic Properties of Hæmatin and Hæmin.*

Having referred to his researches communicated to the Royal Society, in June, 1901, and illustrated the main facts by actual demonstrations, the author discusses (1) observations on the influence of temperature on the behaviour of oxy-hæmoglobin in the magnetic field; (2) observations on the ferro-magnetism of the ferro-albuminates.

(3). *The Specific Conductivity of Solutions of Oxy-hæmoglobin.*

The author next examines the question of the specific conductivity of solutions of pure oxy-hæmoglobin.

In this research he has worked exclusively with the hæmoglobin of the horse, and, following substantially the method of preparation which in Zinoffsky's well-known investigation yielded the purest product, he succeeded in obtaining crystallised oxy-hæmoglobin of remarkable purity, the ash of which consisted solely of oxide of iron with an indeterminable trace of P_2O_5, and contained no trace of chlorine. He employed oxy-hæmoglobin three times re-crystallised, and in addition to the thorough washing by decantation with 20 per cent alcohol after each successive crystallisation, he treated the ultimate product many successive times with large quantities of pure distilled water having a conductivity which never exceeded $10^{-6} \times 2{\cdot}5$, the washed hæmoglobin being separated by the aid of the centrifuge. The solutions which formed the subject of investigation were made by dissolving the mass of moist hæmoglobin crystals in pure distilled water of 35° C., and cooling the solution thus obtained as rapidly as possible.

In his determination of specific conductivities, the author employed the method of Kohlrausch. The bridge was Kohlrausch's metre bridge, of which the platinum-iridium wire was 3 m. in length. This bridge is furnished with resistances of 1, 10, 100, and 1000 ohms, the precision of which had been kindly determined some years ago for the author by Dr. Glazebrook, F.R.S. The resistance vessels employed were those known after the name of Arrhenius, and the temperature was kept constant by immersing them in one of Ostwald's thermostats furnished with a windmill stirrer.

After a laborious investigation on this branch of the subject, the author has arrived at the following conclusions:—

(1). Although solutions of oxy-hæmoglobin possess a low conductivity, this is very much higher than has been found in the previous observations of Stewart, all of which were made at 5° C.

(2). The conductivity of solutions of oxy-hæmoglobin increases rapidly with increase of temperature, and undergoes remarkable and permanent changes when the solution is kept for even short periods at any temperature above 0° C.

These results explain the impossibility of obtaining data which can be considered reliable concerning the *absolute specific resistance* of solutions of oxy-hæmoglobin.

The following numbers expressed in reciprocal ohms represent the mean of the author's results on the specific conductivity of solutions of oxy-hæmoglobin:—

T.	1. Contains 3·07 per cent of O_2Hb (or 1 grm.-mol. in 542900 grms.). Conductivity.	2. Contains 2·235 per cent of O_2Hb (or 1 grm.-mol. in 745800 grms.). Conductivity.
0°	$10^{-5} \times 2{\cdot}626$	$10^{-5} \times 2{\cdot}23$
18°	$10^{-5} \times 4{\cdot}432$	$10^{-5} \times 3{\cdot}25$
25°	$10^{-5} \times 5{\cdot}19$	$10^{-5} \times 4{\cdot}27$
39°	—	$10^{-4} \times 7{\cdot}47$

(4). *The Results of the Electrolysis of Oxy-hæmoglobin.*

1. Continuing the researches contained in his first communication to the Royal Society on this subject, the author finds that when pure solutions of oxy-hæmoglobin are subjected to electrolysis, there occurs a separation of oxy-hæmoglobin in a colloidal but perfectly soluble form. He has worked with currents of from 12 to 24 volts, and the intensity of the electrolysing current measured by a milliampère-meter placed in the circuit has varied in different experiments between 0·1 and 3·0 milliampères.

2. By employing an electrolytic cell in which the anode is separated from the kathode by an animal membrane (sheep's intestine or pig's bladder), it is seen that the first action of the current is to cause a separation of colloidal hæmoglobin in the anode cell. This colloidal hæmoglobin falls as a beautiful red cloud, leaving a perfectly colourless, supernatant liquid. On stirring it instantly dissolves.

3. The further action of the current is to cause a rapid and entire transfer of the colloidal hæmoglobin from the anode to the kathode cell. With an electrolytic cell, of which each compartment had a width of 5 m.m. and contained 2·5 c.c. of a 1 per cent solution of O_2Hb, complete precipitation and transfer occurs within sixty minutes.

4. On reversing the direction of the current by means of a commutator, the hæmoglobin returns again in the direction of the positive current into the original cell from which it started.

5. The author adduces evidence which proves that the precipitated colloidal, but yet perfectly soluble, hæmoglobin represents the undecomposed molecule of the blood colouring matter.

6. The probable nature of the process which occurs under the influence of the current is discussed, as well as the character of the process which leads to the transfer of the hæmoglobin in the direction of the positive current. This process the author considers to be of the same nature as the phenomena studied by Quincke under the name of electro-endosmose.

7. The author directs special attention to the importance of the facts which he has elicited in reference to the colloidal yet soluble form of oxy-hæmoglobin. He points out that all which has been said with regard to oxy-hæmoglobin applies to CO-hæmoglobin.

A typical colloid in the sense of its absolute indiffusi-

bility through animal membranes and parchment paper, oxy-hæmoglobin differs, however, from most colloids in the facility with which it crystallises. Hitherto we have known it in its crystalline condition and in solution in water. Now in its third or colloidal form the analogy with such a colloid as silicic acid is rendered complete.

The discovery of this form of hæmoglobin enables us to form a conception of the state in which the blood colouring matter is probably contained in the blood corpuscles. We have known that the amount of hæmoglobin contained in the corpuscles is so large that in most animals at least the whole of the water of the blood would not be sufficient to dissolve it. It was perfectly obvious, therefore, that it did not exist in the corpuscles in a state of solution, and the opinion has generally been held that these contained some unknown compound of oxy-hæmoglobin with a constituent of the stroma. It seems highly probable that in the red blood corpuscle hæmoglobin may be merely present in its colloidal form.

Finally, the author points out that the remarkable facility with which the new colloidal form of hæmoglobin traverses such permeable membranes as the animal membranes and even parchment paper, when its solutions are subjected to electrolysis, suggests to physiologists the possibility that certain of the phenomena of absorption in the animal body may be closely connected with electromotive changes in the tissues concerned.

EXPLOSION BY HEAT, AND THE DISRUPTION OF BODIES INDEPENDENTLY OF THEIR CHEMICAL COMPOSITION.

By Dr. T. L. PHIPSON.

Shortly after the publication of my little work, "Meteors, Aërolites, and Falling Stars," which appeared in 1867, I made some experiments which led me to discover the cause of the explosion of meteorites when they pass into our atmosphere; and in 1869 I published a short note in the *Comptes Rendus* of the Paris Academy, "*Sur l'explosion et la chute des Météores*," in which I showed that when the surface of any body is *suddenly* raised to an intense degree of temperature, its interior being excessively cold, an explosion invariably occurs.

I alluded again to this in my recent work, "Researches on the Earth's Atmosphere," p. 67 (London: Griffin and Co., 1901), where I have written:—"The cause of the explosion of a meteor in the atmosphere is due to the *sudden rise of temperature on the surface*, which is thereby fused, whilst the interior is most intensely cold. . . . It has been estimated that if the speed of a meteor is only slackened by $\frac{1}{100}$th during its passage through the air, its temperature is raised on the surface to a much higher degree than that at which iron burns; and I have proved by actual experiment that the *sudden* heating of the *surface* of a body intensely cold in its interior, as a meteor coming from cosmic space must be, is quite sufficient to explain the explosion and rupture of the aërolite."

The disastrous explosion of chlorate of potash which occurred not long ago at St. Helens, has given rise to much discussion, and this month to a paper by Professor Dupré, read to the Society of Chemical Industry, in which he shows that beads of chlorate of potash exposed suddenly to a very intense heat will explode even when no reductive gas is present; and in the discussion which followed the reading of that paper, it was shown that M. Berthelot had already arrived at the same opinion shortly before. But I made a similar experiment in 1869, and arrived at this opinion thirty-three years before either Prof. Dupré or M. Berthelot.

My first experiment on this subject was a very simple one; I found that a substance which, when heated gradually, simply ignites, without any explosion whatever, when *suddenly* exposed to the hottest part of a furnace at a white heat, invariably explodes.

I therefore consider myself justified in laying claim to the discovery of this phenomenon until any chemist can prove that he made the experiment I made previous to the year 1869.

It is evident that my little note in the *Comptes Rendus* for that year clearly pointed out that there is a hitherto unrecognised property in what we call *heat* or *caloric*, which causes in certain circumstances the explosion of bodies quite *independently of their chemical composition*.

Probably the same molecular force which, when acting slowly, produces what we call *dilatation* when acting in the above-mentioned circumstances, produces what is termed *explosion*. It is a subject which requires much further investigation, and may possibly lead to some modifications of the present theory; but, personally, I am no longer able to carry on such investigations.

Casa Mia, Putney, March 22, 1902.

THE ANALYSIS OF TIN ORES.

By J. A. MULLER.

The different recognised methods for the estimation of tin do not, as a rule, give entire satisfaction, in so far as the exactness of the results obtained is concerned. The reduction of cassiterite, digested in aqua regia, by fusing with cyanide and weighing the metallic mass obtained after washing, drying, and fusion under a layer of stearic acid, only gives good results when the ore is approximately pure. But in the presence of inert bodies, such as quartz, the reduction of the powdered ore gives a very divided metallic residue, which cannot afterwards be collected into one button or ingot when melted under stearic acid.

The fusion of the ore with a mixture of carbonate of potash and sulphur gives a similar result. The attack is only complete with the pure binoxide; but, in the opposite case, the residue must be submitted to three or four successive treatments for the whole, or nearly so, to be extracted in the form of a sulpho-salt soluble in water,

To estimate the tin in cassiterite, W. Hampe (*Chem. Zeit.*, 1887, p. 19) weighs the pulverised ore in a porcelain boat and reduces it by heating for two hours in a current of dry hydrogen; after which he dissolves the tin formed in hydrochloric acid and estimates it in the usual manner.

It is clear that if the ore only contains binoxide of tin as the substance reducible by hydrogen, the difference in the weight of the boat before and after the reduction will enable us, if the reduction is complete, to immediately calculate the weight of tin contained in the substance under examination; and, further, this method has the advantage of being very rapid, for it is quite possible to effect a large number of reductions in the same tube at the same time.

I have endeavoured to decide what was the necessary and sufficient time for the reduction of binoxide of tin, by heating to a moderate red heat, for different times, known weights of pulverised cassiterite, either mixed or not with quartz, in a current of pure dry hydrogen, taking good care to regulate the flow of the latter so as always to have a large excess of the reducing gas.

By using 0·9981 grm. of cassiterite, previously digested in aqua regia, and containing 97·47 per cent of SnO_2 and 2·31 per cent of quartz, I found the loss of weight to be 0·2007 grm. after heating for two hours in hydrogen, and 0·2062 grm. after three hours; now theory gives 0·2068 grm. for the total reduction. With a mixture of 0·5334 grm. of cassiterite and 0·3282 grm. of quartz, the loss of weight was 0·1050 grm. after two hours, and 0·1102 grm. after three hours and a half; theory gives 0·1105 grm.

The preceding experiments show that it requires at least three hours to effect the total reduction of the stannic oxide.

The following experiments—of which the first two were made with the cassiterite previously employed, and the three last with a cassiterite containing 99·08 SnO_2, 1·10 SiO_2, 0·10 CuO, and traces of oxide of iron and zinc—show that three hours is sufficient in all cases:—

Mixture.		Loss of weight.		Calculated loss of weight.	Tin, per cent.	
Cassiterite.	Quartz.	After 3 hours.	After 4½ hours.		Found.	Calculated.
Grm.	Grm.	Grm.	Grm.	Grm.		
0·8118	0·2108	0·1690	0·1689	0·1682	61·20	60·92
0·4517	0·6167	0·0941	0·0940	0·0936	32·62	32·45
0·2354	0·7932	0·0490	—	0·0496	17·64	17·85
0·9757	—	0·2054	—	0·2055	77·96	78·01
1·5363	—	0·3242	—	0·3236	78·14	78·01

Thus, whether the binoxide of tin is mixed or not with inert bodies, the loss of weight in hydrogen enables us to estimate with a pretty fair approximation the percentage of tin in the substance analysed.

According to "Würtz's Dictionary," the different minerals which are likely to be met with in the deposits of cassiterite are quartz, mica, topaz, fluor-spar, apatite, blende, galena, mispickel, molybdenite, and wolfram.

Quartz, muscovite, and topaz do not undergo any change of weight when heated in hydrogen under the conditions of the above experiments. Of this I have made quite certain; they therefore do not interfere with the operation in any way. As to the other minerals, they can be removed by treating the powdered mixture successively, and several times, with hot aqua regia, hot water, a neutral solution of tartrate of ammonium (to eliminate a little sulphate of lead which might be present), and, finally, with ammonia and water to dissolve the molybdic and tungstic anhydrides.

It is true that this separation offers practical difficulties, especially on account of the filtrations it is necessary to make *only* after complete deposition of the solid matters, as the pulverised bodies in suspension will pass through the best filter-papers.

Cassiterite loses practically nothing of its weight when heated with aqua regia. Thus, 1·9564 grms. of calcined cassiterite, previously submitted to the preceding series of treatments, were heated for three days at about 50° with 12 to 15 c.c. of aqua regia (2 volumes of fuming HCl and 1 volume of HNO_3 at 36° B.), the liquor being renewed every day. An insoluble residue was left which, after calcination, weighed 1·9558 grms.; in the solution I found 0·4 m.grm. of SnO_2 and 0·3 m.grm. of Fe_2O_3.

To make quite certain up to what point we could count on the exactness of the results obtained in the particularly inconvenient case of a mixture containing galena and wolfram, a mixture was made of the three following finely powdered minerals:—Cassiterite at 99·08 Sn, 0·5859 grm.; galena,* 0·4532 grm.; and wolfram,† 0·3993, which I submitted three separate times to the series of treatments already described. Finally, the residue, previously calcined at a red heat, was submitted to the action of hydrogen. The loss of weight was 0·1239 grm., which corresponds to 31·9 per cent of tin in the mixture, while there was actually 31·78 per cent; the agreement is thus very satisfactory.

After heating the digested mineral in hydrogen, the residue may be treated with fuming hydrochloric acid and the solution analysed by weighing the tin in the form of binoxide, after precipitation as sulphide, treating with sulphide of sodium, &c.; it is in this manner that the cassiterites used in this research were analysed.

I have endeavoured to apply the above-described method of estimating tin, applicable to cassiterite, to stannine, which is a sulphurised ore of tin.

For this purpose the pulverised stannine was calcined at a dull red heat in a muffle; then, after treating the residue with a little nitric acid, it was dried and re-calcined; then treated again with nitric acid, dried at 110°, and digested first with dilute nitric acid, then with aqua regia. The residue thus obtained should consist entirely of binoxide of tin, quartz, and silicates, which are not attacked by the above treatment. As a matter of fact, this residue always contains a small quantity of ferric oxide, which I have never succeeded in completely eliminating, even by repeated treatments with aqua regia. Thus, in one experiment, 1·4414 grms. of stannine gave a residue which weighed, after calcination at a red heat, 0·4865 grm., and of which 0·4837 grm. lost 0·1036 grm. in hydrogen; this corresponds to a proportion of tin equal to 26·77 per cent. Now, the examination of the different solutions resulting from the treatment of the calcined stannine, and the analysis of the residues after the action of the hydrogen and treatment with hydrochloric acid, has given me the following figures for the composition of this stannine, supposing all the metals were in the form of sulphides:—

	I.	II.	III.
SnS_2	38·48	38·36	38·48
Cu_2S	33·72		
FeS	16·87		
ZnS	8·55		
Quartz	1·45		
	99·07		

This stannine therefore contained 24·94 per cent of tin, while the estimation quoted above gave 26·77 per cent.—*Bull. Soc. Chim.*, Series 3, xxv., No. 23.

MOUNT VERNON LOESS.

Loess occurs to a considerable depth on the hills in the vicinity of Mount Vernon, Iowa. On the somewhat elevated ridge on which Cornell College is located, the formation extends to a depth of 40 feet, diminishing from the summit. In general it overlies the Kansan and Paha drifts, but it is usually absent over the Iowan.

A brickyard is located in the Mount Vernon Loess from which a good quality of brick is obtained.

The specimen chosen for analysis was taken from the brickyard, 8 feet below the surface of the earth.

The analysis was made by Frank Hann, in the Chemical Laboratory of Cornell College, under the direction of Dr. N. Knight.

The following results were obtained:—

	Per cent.
SiO_2	70·86
CO_2	4·70
Fe_2O_3	2·97
Al_2O_3	8·91
MnO_2	0·28
CaO	4·13
MgO	3·12
K_2O	1·18
Na_2O	1·69
TiO_2	0·59
P_2O_5	0·40
FeO	0·10
H_2O	1·10
	99·98

* After treatment by aqua regia, &c., 1·671 grms. of this galena left a residue weighing 0·0586, which lost none of its weight at a red heat in hydrogen.

† A beautiful sample, of which the density was 7·13, and which gave the following analysis:—

WO_3 sol. NH_3	74·67 per cent
Insoluble part NH_3	1·22 ,,
FeO	11·07 ,,
MnO	12·12 ,,
CaO	1·26 ,,
	100·34

THE ACTION OF BINOXIDE OF SODIUM ON THE METALS OF THE PLATINUM GROUP.

By MM. LEIDIÉ and QUENNESSEN.

THE reactions given in text-books on analytical chemistry for the salts formed by chromium, cobalt, and the six metals of the platinum group, apply to the *salts* of these metals. In the case of the *ammoniated* derivatives, these reactions are no longer applicable; it is necessary to decompose the compound, and make the test for the metal on the metal itself. When we have to deal with chromium and cobalt the experiment is easy; but in the case of the metals of the platinum group, many and complicated operations have to be gone through which necessitate the use of numerous samples of the material.

The authors have succeeded in effecting this analysis in one operation only, carried out on one sample of material, by means of a new method—the oxidation of the metal by binoxide of sodium under the influence of heat.

The metal, in the form of fine powder, is mixed with five or six times its weight of binoxide of sodium, and the mixture, placed in a nickel crucible, is heated gently over a gas flame until the mass, stirred from time to time with a nickel spatula, becomes semi-fluid. It is then allowed to cool, and the product of the reaction is put into cold water (about ten times the weight of the binoxide); this must be done carefully on account of the evolution of oxygen which takes place through the decomposition of the excess of binoxide, and also on account of the rise of temperature due to the hydration of the oxide of sodium. The mixture is poured into a beaker, and allowed to settle; the liquor is decanted after complete deposition, and the precipitate is thrown on a filter, after having added water; this is done to prevent the filter being pierced by the too alkaline liquor. We thus get, on the one hand, a solution, and, on the other, a precipitate; the first serves for the search for osmium, ruthenium, palladium, and indium, the second for platinum and rhodium.

The following is a summary of the experimental results obtained by the authors. This summary will explain the systematic table prepared by the authors for the plan of analysis:—

The *osmium* is integrally transformed into osmiate of sodium, which dissolves in water, giving a yellow solution. Under the influence of a current of chlorine, peroxide of osmium, OsO_4, is given off; this is collected in a vessel containing iced water; it is recognised by the action of hot nitrite of potassium, which forms violet osmiate of potassium, which is precipitated.

The *ruthenium* is integrally transformed into ruthenate of sodium, soluble in water, giving an orange-yellow coloured solution; this solution, treated with chlorine in the same manner as the one containing osmium, gives peroxide of ruthenium, RuO_4, which is recognised by the action of hot hydrochloric acid with the formation of a soluble sesquichloride of ruthenium of a dark brown colour.

The *palladium* is transformed into palladate of sodium, which colours the solution yellow; this latter is evaporated after having been neutralised by hydrochloric acid, and treated with a little aqua regia; with chloride of potassium it gives a ruby-red precipitate of chloropalladate, $PdCl_4 2KCl$, insoluble in saturated solutions of chloride of potassium.

The *iridium* gives a basic iridate of sodium, which colours the solution blue; this, treated in the same manner as the palladium solution, gives black crystals of chloroiridate, $IrCl_4 2KCl$, insoluble in saturated solutions of chloride of potassium.

The *platinum* forms a platinate of sodium, insoluble in water, but soluble in hot hydrochloric acid; it is thus found in the precipitate. This latter is treated with hot hydrochloric acid to dissolve it. The solution is evaporated after having been neutralised by hydrochloric acid, and treated with a little aqua regia; with chloride of potassium it gives a ruby-red precipitate of chloroplatinate, $PtCl_4 2KCl$, insoluble in saturated solutions of chloride of potassium.

We thus obtain:—

A. A solution.
B. A precipitate.

The product resulting from the attack of the metal with binoxide of sodium is treated with water.

A. The solution is coloured.

More or less deep *yellow* colour. Submitted to a current of chlorine while hot.

A substance is volatilised which gives a black precipitate with NH_4S.

The distilled solution gives an insoluble violet osmiate with hot KNO_3. No brown coloration with HCl .. .. Osmium.

The distilled solution gives a brown coloration (Rh_2Cl_6) with hot HCl .. Ruthenium

Nothing volatilised. No black precipitate with NH_4S in the distilled liquor.

Neutralise with HCl; evaporate in the presence of HCl with HNO_3 and KCl. *Red* crystals insoluble in HCl. . .. . Palladium.

Blue colour. Neutralise with HCl. Evaporate in the presence of HCl with HNO_3 and KCl. Black crystals, insoluble in KCl .. .. Iridium.

B. The solution is colourless. It is thrown away, and the precipitate treated with HCl. The filtered liquor is treated with NO_2Na, then with CO_3Na_2. Filter.

The filtered solution is treated with excess of HCl and evaporated nearly to dryness. Take up with water.

The solution is of a light *yellow* colour. With NH_4Cl it gives a yellow precipitate insoluble in excess of NH_4Cl .. .. Platinum.

The solution is coloured *red*. It is not precipitated with NH_4Cl .. Rhodium.

porated down to drive off the greater part of the acid, then treated first with nitrite of sodium and then with carbonate of sodium. In this manner the platinum, as well as the nickel resulting from the attack of the crucible, are both transformed into double nitrites; now this salt of nickel is precipitated by carbonate of sodium, while the platinum salt is not; thus on filtration we have a solution containing platinum free from nickel.*

The double nitrite of platinum and sodium is transformed into chloroplatinate of sodium by evaporating down, after the addition of hydrochloric acid in excess. The residue, taken up with water and treated with chloride of ammonium, gives chloroplatinate of ammonium of a yellow colour, and insoluble in an excess of the reagent.

The *rhodium* is transformed into a binoxide, insoluble in water, but soluble in hot hydrochloric acid. The precipitate containing it is treated with hot hydrochloric acid; after filtration the solution is evaporated to drive off the acid, then treated with nitrite of sodium and carbonate of soda, as in the case of platinum; the nitrite of nickel is precipitated by the carbonate of soda, while that of rhodium is not. The solution of this latter, decomposed by hot hydrochloric acid, gives the double sesquichloride, Rh_2Cl_6NaCl, of which the solution is red, and does not give any precipitate with chloride of ammonium.

The results are summarised in the accompanying table, which will serve as a guide for a qualitative analysis.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING FEBRUARY 28TH, 1902.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To CHARLES PERRIN, Esq., M.Inst.C.E.,
Water Examiner, Metropolis Water Act, 1871.

London, March 10th, 1902.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 192 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from Feb. 1st to Feb. 28th inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 192 samples examined by us during the month, all were found to be clear, bright, and well filtered.

The rainfall at Oxford again shows a serious deficit; the actual amount of rain measured was only 0·95 inch, the average fall is 1·80 inches; this makes a deficit of 0·85 inch, and, added to the deficit recorded in January, makes a total shortage of 2·27 inches, or 58·5 per cent on the thirty-five years' average.

Our bacteriological examinations of 485 samples have given the results recorded in the following table; we have also examined 36 other samples, from special standpipes, wells, &c., making a total of 521 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 24 samples) ..	197
New River, filtered (mean of 117 samples) ..	9
Thames, unfiltered (mean of 23 samples) ..	3329
Thames-derived water, from the clear-water wells of eight Thames-derived supplies (mean of 264 samples)	27
Ditto ditto .. .. highest	205
Ditto ditto .. .. lowest	0
River Lea, unfiltered (mean of 24 samples) ..	233
River Lea, from the East London Company's clear-water wells (mean of 33 samples) ..	8

Of the 414 samples of impounded and filtered water supplied to London and examined bacteriologically during the past month, 13 samples, or 3·1 per cent, were sterile. Only two samples contained more than 150 microbes, while only nine samples, or 2·1 per cent, contained more than 100 microbes per c.c. The mean number of microbes in the nine excess samples was 143, against a corresponding mean of 143 in thirty excess samples during January.

It will be seen that, although there has been a smaller number of sterile samples during this month, there has been a still greater decrease in the number of samples containing over 100 microbes, with the satisfactory result that the average has been reduced. This is not to be wondered at, considering that the supply of the River Thames has been drawn largely from the storage in the great chalk deposits, consequent on the very small rainfall during the month in the valley of the Thames.

We are, Sir,
Your obedient Servants,
WILLIAM CROOKES.
JAMES DEWAR.

ON PYRITE AND MARCASITE.*

By H. N. STOKES.

(Concluded from p. 136).

XII. CONSTITUTION OF PYRITE AND MARCASITE; ACTION OF CUPRIC SALTS (*continued*).

Experiment with Cupric Chloride and Pyrite.

Experiment 6.—0·2545 grm. pyrite was heated with 50 c.m.³ of a pure cupric chloride solution containing as much copper as the sulphate used in the foregoing experiments; the tube being filled with the usual precautions to prevent oxidation. The tube was heated five hours at 200°, and on cooling was found to contain undecomposed mineral and an abundant crystallisation of cuprous chloride. The filtered solution gave—

0·0667 grm. iron = 0·1429 grm. pyrite.
0·5540 grm. $BaSO_4$ = 0·1427 grm. pyrite.

It appears, therefore, that as far as it was decomposed the pyrite was completely oxidised to ferrous salt and sulphuric acid, with a corresponding reduction of the copper from the cupric to the cuprous state, according to the equation—

$$FeS_2 + 14CuCl_2 + 8H_2O = 14CuCl + FeCl_2 + 2H_2SO_4 + 12HCl.$$

I have further obtained an abundance of sulphuric acid by

* All metals, without exception, are attacked by binoxide of sodium under these conditions. We have chosen nickel (chemical dishes, &c., made of this metal are easily obtained), firstly, because its oxide is insoluble in water, which fact enables us to separate it from osmium, ruthenium, palladium, and iridium, and also because its double nitrite of sodium is precipitable by the alkaline carbonates, while the double nitrites of platinum and rhodium are not; this enables us to separate the platinum and rhodium. It is one of the general properties of the double nitrites of the metals platinum and sodium not to be precipitated by the alkaline carbonates. This is the basis of the general method devised by M. Leidié for the extraction and separation of the metals of the platinum group. See E. Leidié, *Comptes Rendus*, vol. cxxxi., p. 888; *Bull. Soc. Chim.*, [3], vol. xxv., p. 9; *Journ. Pharm. et Chim.*, [6], vol., xiii., p. 18.

* From the *Bulletin of the U.S. Geological Survey*, No. 186.

oxidising precipitated copper sulphide with cupric chloride solution at 100°, air being rigorously excluded. Doubtless in the above experiments with pyrite and marcasite and copper sulphate an analogous oxidation occurs, being obscured by other reactions. Chalcocite, however, is not perceptibly attacked by 10 per cent copper sulphate solution after fourteen hours' heating at 200°.

Experiment with Ferrous and Cupric Sulphates.

Experiment 7.—A mixture of equal volumes of 10 per cent solutions of crystallised copper sulphate and crystallised ferrous sulphate (carefully freed from ferric salt by reduction with hydrogen sulphide) was heated, under absolute exclusion of air, for three hours at 200°. A considerable red precipitate was obtained, which consisted of a mixture of ferric oxide or basic sulphate with cuprous oxide and apparently metallic copper. The reduction of cupric by ferrous salts in neutral or acid solution is frequently referred to in the literature, but, so far as I can ascertain, without experimental basis.

Theory of the Reaction.—The explanation of the result is probably the following:—In the mixture we have equilibrium between the cupric, cuprous, ferrous, and ferric ions and copper, as represented by the equations—

$$Cu'' + Fe'' \rightleftarrows Cu' + Fe'''$$
$$Cu'' + 2Fe'' \rightleftarrows Cu + 2Fe''',$$

in which but traces of Cu, Cu', and Fe''' exist. (The slight solubility of cuprous compounds shows that not more than traces can be present). On heating, the trace of ferric salt is precipitated in the basic form, the equilibrium is disturbed, and the reaction continues to proceed from left to right, accompanied by formation of Cu' and Fe''' (and possibly Cu) and their precipitation as insoluble products, until the accumulated acid is in equilibrium with both of these and further precipitation cannot occur. The process is further complicated by a partial precipitation of copper as basic salt when the sulphate is used. A mixture of cupric and ferrous salts gives a strong reaction for ferric iron with sulphocyanate. This does not imply an appreciable oxidation of the ferrous iron in the mixture itself, but a shifting of the equilibrium to the right in the above equation through the formation of the slightly dissociated ferric sulphocyanate. It is doubtless to the reduction of the cupric sulphate by sulphur and by ferrous sulphate that the formation of chalcocite from pyrite is to be ascribed.

General Conclusions from the Experiments.

The conclusions which may be drawn from the above experimental data are:—

1. At 200° copper sulphate decomposes marcasite more rapidly than pyrite.

2. Both ferrous and ferric salts are formed in both cases, the former being found wholly in the solution, the latter wholly in the precipitate. There is no marked difference in the relative amounts of these in either case. The precipitate also contains, besides cuprous sulphide, cuprous oxide and perhaps metallic copper.

3. The formation of ferric salt is to be ascribed, in part at least, to the reaction of the ferrous salt upon the cupric salt (Experiment 7), while some oxidation of sulphur to sulphuric acid occurs (Experiment 6). Any ferric salt which may fail to be precipitated will be reduced by the sulphides.

4. The relative amounts of ferrous and ferric salts depend simply upon the establishment of equilibrium between the solution and the products of decomposition of the pyrite or marcasite, and not upon any fundamental chemical difference in the two minerals, and consequently the hypothesis of Brown is devoid of valid experimental basis.

It would seem that no method can be depended upon to give us an insight into the state of combination or valency of either iron or copper in their sulphides which does not take due account of the conditions of equilibrium between the reagents and the decomposition products. This applies not only to other experiments of Brown quoted in his paper, but to the attempt of Starke, Shock, and Smith (*Journ. Am. Chem. Soc.*, 1897, vol. xix., p. 948) to prove the divalent condition of iron in arsenopyrite, and of Morgan and Smith (*Journ. Am. Chem. Soc.*, 1901, vol. xxiii., p. 107) to establish the same for chalcopyrite. That the product of heating chalcopyrite in dry hydrochloric gas consists of a mixture of cupric chloride and ferrous chloride is inconclusive on the question as to whether chalcopyrite has a constitution represented by $(Cu''Fe'')S_2$ or $(Cu'Fe''')S_2$ until the condition of the system $CuCl + FeCl_3 \rightleftarrows CuCl_2 + FeCl_2$ in equilibrium is known, in the absence of water and presence of hydrochloric acid gas. That nearly all the iron was obtained in the ferrous state in some of the experiments with copper sulphate made by Brown and others I can explain only on the assumption that enough acid was originally present or eventually formed to enable any basic salt to act on and be reduced by the residual sulphides and cuprous oxide. In fact, hot dilute sulphuric acid slowly dissolves the mixture of cuprous oxide and basic ferric salt obtained in my experiments, giving a strongly reducing solution.

XIII. Oxidation of Pyrite and Marcasite by Potassium Permanganate.

(For previous experiments on the action of permanganate on pyrite and marcasite see A. P. Brown (*Proc. Am. Phil. Soc.*, 1894, vol., xxxiii.; and Chemical News, 1895, vol. lxxi., pp. 131, 155, 171). The experiments of Brown show that, with an excess of permanganate, the total sulphur oxidised in a given time is greater in the case of marcasite than of pyrite, but they do not establish any relation between the amount of sulphur oxidised and the quantity of mineral decomposed).

A few experiments were made to determine the percentage of sulphur oxidised in pyrite and marcasite at ordinary temperature by acidified permanganate, the mineral being always in excess.

An excess of the powdered mineral, freed from oxidation products, was shaken in a stoppered cylinder with a constant amount of dilute sulphuric acid and 100 c.m.^{3} $KMnO_4$, added in quantities of 5 c.m.^{3} at a time, the shaking being continued after each addition until the colour had disappeared, which required but a few minutes. The filtered solution was then reduced and the iron titrated by the same permanganate solution.

The proportions required are—

For complete oxidation to $Fe_2(SO_4)_3$ and sulphuric acid	$5FeS_2 + 15KMnO_4$
For oxidation of the iron alone to $Fe_2(SO_4)_3$	$5FeS_2 + 3KMnO_4$
For oxidation of the sulphur	$12KMnO_4$

Let—

a = volume of permanganate used in oxidation of FeS_2,

and—

b = volume of permanganate required to re-oxidise FeO to Fe_2O_3;

then—

$3b$ = volume of permanganate used in oxidising Fe in FeS_2 to Fe_2O_3,

and—

$a - 3b$ = volume of permanganate used in oxidising S in FeS_2 to SO_3;

also—

$12b$ = volume of permanganate required to oxidise all the sulphur in the mineral decomposed;

whence—

$$\text{Percentage of sulphur oxidised} \ldots = p = \frac{100\,(a - 3b)}{12b} = \frac{8{\cdot}333a}{b} - 25.$$

Where 190 c.m.³ of permanganate are used,—

$$p = \frac{833{\cdot}3}{b} - 25.$$

The values of p obtained were—

	1.	2.
Pyrite	94·0	90·7
Marcasite..	84·6	86·7

The duplicate results are therefore not sufficiently concordant, nor is the difference between pyrite and marcasite great enough, to give the method more than a confirmatory value. The difference here, as in the case of oxidation by the ferric sulphate method, is due to the greater ease with which marcasite is attacked, but the relative oxidation rate of the sulphur is clearly much greater. The difference might possibly be greater at 100°, but here the experiments failed because of the strong tendency of the permanganate to deposit peroxide, even in the presence of much sulphuric acid.

XIV. Summary of Results.

The chief results of this investigation may be thus summarised:—

1. When pyrite or marcasite is boiled with an excess of a solution of ferric salt to complete reduction of the latter, the ratio of sulphur oxidised to mineral decomposed is perfectly definite and characteristic of each mineral, provided certain standard and easily controllable conditions are observed. Under these conditions the percentage of sulphur oxidised in pyrite is about 60·4 per cent and in marcasite about 18 per cent of the total sulphur. These figures are the characteristic *oxidation coefficients*.

2. The oxidation of pyrite or marcasite to ferrous salt, sulphuric acid, and free sulphur cannot be expressed by any single equation, but takes place according to two or more, the relation between which varies with the special conditions.

3. An empirical curve for the oxidation coefficients of mixtures of pyrite and marcasite is constructed by aid of which the composition of naturally occurring mixtures may be quantitatively determined.

4. The influence of various impurities on the results is described.

5. Various concretions and other specimens are examined, and it is shown that in many cases much uncertainty exists in distinguishing pyrite and marcasite by the usual methods.

6. There is no well-established evidence of the existence of true paramorphs of pyrite after marcasite or of marcasite after pyrite.

7. The hypothesis that most natural specimens, even when well-crystallised, are intimate mixtures of the two is without foundation.

8. Specimens crystallising in the regular system are true pyrite, while those forming rhombic crystals are true marcasite. When the two are mingled or intergrown, it is generally possible to distinguish each by the colour after cleaning with acid—a rule to which there are a few exceptions.

9. The density does not afford a trustworthy means of determining one mineral in the presence of the other.

10. Chalcopyrite may be sharply distinguished from chalcocite or bornite in pyrite carrying 3 per cent copper or less. A method is given for detecting small amounts of chalcopyrite in pyrite and marcasite, or the reverse, or in rocks.

11. There is no evidence as to the state of combination or valency of iron in pyrite and marcasite, or that these differ in the two minerals. Experiments which have been regarded as proving the existence of such differences are inconclusive.

12. The principle of the oxidation method, with appropriate modifications, is probably capable of wider application in distinguishing dimorphous minerals, in determining whether a given complex mineral is a mixture or compound of its simpler constituents, and in determining the nature of small amounts of impurities.

13. The action of acid permanganate on pyrite and marcasite is analogous to that of ferric salts, the percentage of sulphur oxidised in pyrite being higher than in marcasite.

My thanks are due to Dr. A. A. Julien, whose valuable assistance has been noted above, and to Professors Van Hise, Emerson, Merrill, and Penfield, and Messrs. Tassin, Emmons, Ransome, Weed, and Lindgren, who have generously supplied most of the material upon which the foregoing investigation is based.

NOTICES OF BOOKS.

The Elements of Physical Chemistry. By J. Livingston R. Morgan. Second Edition, Revised and Englarged. New York: John Wiley and Sons. London: Chapman and Hall, Ltd. 1902. Pp. x.-352. 12mo.

Since publishing the first edition of this book noticed in Chemical News, vol. lxxix., p. 129 (March 17, 1899), the author has done three things to improve it; he has brought the subject-matter itself up to date, he has made the relations clearer than before, wherever possible, and he has endeavoured to make the volume more useful to those studying the subject without an instructor. A few things found to be superfluous have been omitted, and a number of new relations have been added; also 150 problems.

This comprehensive outline of subjects that have been treated in independent volumes is so arranged as to economise the time that the average student can spare for this branch of chemistry; at the same time those desirous of extending the study of any special portion can do so with the aid of the references given. The calculus is introduced in devising some of the laws, but most of the work requires no special training in the higher mathematics.

Those wishing to pursue the study of physical chemistry along the lines of modern research, especially with reference to solution, thermochemistry, the laws affecting chemical change, and electrochemistry, will find this useful manual generally up to date and reliable. The subject of phases is but briefly treated, and reference is made to the special treatise by Wilder Bancroft, "The Phase Rule."

The author lays no claim to originality, but acknowledges his indebtedness to Ostwald, to Le Blanc, and to Nernst. The book is well printed, and has an index. H.C.B.

Manual of Alcoholic Fermentation and the Allied Industries. By C. G. Matthews, F.I.C., F.C.S. London: Edward Arnold.

This book contains, amongst others, chapters on the life-history of the saccharomycetes, chemical science and the carbohydrates, the preparation of wine, and the brewing process. From these titles it will be seen that its scope is fairly large, and that it is intended to appeal to a wide circle of readers; probably it would have been wiser to have restricted its aim.

At the very outset exception must be taken to the definition of fermentation, and the review of the present state of our knowledge of the theory of fermentation is not entirely up-to-date. The full significance of Buchner's work on the isolation of zymase, and the essential difference between regarding fermentation as a vital metabolic process of the yeast, and assuming the presence of an enzyme produced by the yeast cells, seem to have been hardly realised by the author. Much of the chapter on chemical science either requires fuller treatment, or had better have been omitted altogether; both the student and technologist might well be expected either to have

a previous knowledge of chemistry, if only of an elementary nature, or at least to be able to acquire it from other sources. Moreover, the accuracy is not always beyond dispute, nor the wording happy. For the practical man details of the brewing process would probably be insufficient, and no mention is made of the diseases of beer, though those of wine are briefly touched upon; the brewery plant, too, might well have been described in fuller detail. A word of praise must be given to the plates and illustrations, which are very clear and good.

Die Wissenschaftlichen Grundlagen der Analytischen Chemie. By W. Ostwald. Leipzig: Wilhelm Engelmann. 1901.

The aim of this book is expressly stated to be not the teaching of analytical chemistry as such, but the explanation of the theoretical groundwork underlying analysis; hence, with this object in view, few practical details are to be found in it, while no theoretical point is overlooked. The book is characterised by Dr. Ostwald's well-known thoroughness of treatment in regard to details, too often hurriedly passed over, or altogether neglected in elementary text-books, such as, for example, the theory of the washing of precipitates and of distillation. As we should expect from so staunch a champion of the ionisation theory, the subject is attacked from an ionic standpoint. Beginning with purely theoretical considerations, the first part deals with the recognition and separation of substances in general, both by physical and chemical means, and ends with a short account of the theory of methods of quantitative separation. Dr. Ostwald's style makes this part of the book particularly attractive reading. The second part contains, under the headings of the several elements, the theory of the application of general principles; methods of separation are not neglected, but no tables for detection and separation are given. In the appendix a few experiments illustrative of the text are briefly described, admittedly by no means sufficient in number; the amplification of these, however, may well be left to the choice and discretion of the teacher.

The book has well deserved its success, but we should have expected to see a third edition quite free from even trifling misprints, and an index would probably be appreciated by those who may have occasion to refer constantly to the book.

Ferments and their Actions. By Carl Oppenheimer, M.D., Ph.D. Translated from the German by C. Ainsworth Mitchell, B.A. (Oxon.), F.I.C. London: Charles Griffin and Co., Ltd. 1901.

The translation of this thoroughly scholarly treatment of a wide and difficult subject deserves a good reception in England; the translator's work has been well done, and in some instances he has, with the author's sanction, introduced the results of investigations published since the appearance of the German edition; we could wish—though of course this is a minor point—that italics had been a little more sparingly used.

The word ferment is used in its widest sense, though putrefactive processes and the so-called butyric acid fermentation are omitted. An exceedingly cautious definition of a ferment provides a subject for masterly chapters on the conception of the nature of a ferment, and the chemical nature of ferments, in which various hypotheses are fairly discussed, even if ultimately dismissed as untenable. The ferments are classified under the headings hydrolytic and oxidising; for the present, lactic acid fermentation is included with the former, perhaps without very good reason. Every effort has been made to give the results of the most recent researches, a very essential point in such a work; so rapidly is our knowledge of physiological chemistry growing, however, that every month sees a change in our views; for instance, the action of trypsin on pepsin, contrary to our author, has been recently shown to be not altogether negligible.

The whole is well indexed and arranged, and liberal acknowledgments are everywhere made, and references given to original papers, the author believing firmly in the necessity for reference to original publications for experimental details.

Water and its Purification; a Hand-book for the Use of Local Authorities, Sanitary Officers, and Others interested in Water Supply. By Samuel Rideal, D.Sc. (Lond.), F.I.C., &c. With numerous Illustrations and Tables. Second Edition. Revised and Extended. London: Crosby Lockwood and Son. 1902. Pp. 346.

Pure water, as the term is generally understood, is very different to really pure water from the chemical point of view. The best water for human use should contain a variety of substances held in solution, such as salts of lime, sodium, &c., as well as gases, such as carbonic acid and free oxygen, all of which help to make it palatable. There are, however, other substances which should always be kept out of water as far as possible when it is intended for dietetic purposes.

The main object of this book is to give some insight, to those interested in the subject of water supply, into the methods of research as to the quality of various waters, and the interpretation of the results obtained. Although it is impossible for any but a trained expert to interpret the results of analyses with accuracy, it often happens that a report might be made more easy of comprehension to an ordinary reader; at the same time it must be insisted upon, that the so-called rough and ready tests are so likely to be misleading as to be practically valueless.

The ordinary suspended impurities to be found in raw waters may be divided into three classes, viz., vegetable, animal, and mineral; practically all these can be removed by efficient filtration, and even germs of disease may be so few in number as not to be found in every sample; on the other hand, chemical products being dissolved will be found throughout. These products exist in great variety, and form a good index of the permanent character of the water.

Experience has shown that sand filtration is not only the best means for removing suspended matters on the large scale, but also, through the formation of the surface slime on the filter, for the oxidation of the organic matter dissolved in the water by the combined help of the microbes present and the oxygen held in solution. A sand filter does not attain its maximum efficiency until this slime has been formed. After a time, however, the growth increases to such an extent that the bed becomes clogged, and it becomes necessary to renew the whole of the surface layer.

It not infrequently happens that a water contains a considerable amount of lime and magnesia salts; it is known as hard water, and, besides not being so good for domestic use, it is very unfit for manufacturing purposes. It then becomes necessary to soften it; one of the best known methods for effecting this is Clarke's process; this and other methods are dealt with very fully in Chapter X.

The chapter on analysis and its results is of interest, but the subject is only lightly dealt with. The combustion process of Frankland and Armstrong is accorded only a few lines more than a page, and then only to point out a few possible sources of error. We must take exception to the author's remarks on this method of determining the organic carbon and nitrogen in water, as every one of these objections indicates either lack of knowledge or lack of skill in carrying out the process. We admit that the process is not simple, like the estimation of the organic matter in solution by means of permanganate of potash, but in the hands of a careful and well-trained chemist, trustworthy results can be obtained with remarkable

accuracy, and not only the amount but the character of the organic matter is also shown, whether of animal or of vegetable origin, which cannot be done with any degree of certainty by any other method.

The appendix deals with some recent epidemics connected with water supply, and suggestions for legislation in connection therewith. There are some useful tables giving typical examples of water analyses, the composition of boiler incrustations, the order of the rocks, and characteristics of waters derived from them, &c., while the index, which fills twenty-six columns, is exceptionally complete.

Property in Trade Marks. Being a Manual Written, without the Use of Legal Phraseology, on Trade Marks and their Protection. By Brewer and Son, Patents, Designs, and Trade Marks Agents. London: Taylor and Francis. 1902. Pp. 78.

The ordinary works on the subject of trade marks are written in such highly technical language that, though they are of great value to specialists who have made a study of the subject, they are of little or no use to the manufacturer or trader who wishes to arrive at a clear understanding of the law relating to property in trade marks.

The object of this book is to enable the users and prospective users of trade marks to glean some knowledge of the intricate law governing this matter; and it is also suggested that patent agents and solicitors, who only occasionally have to deal with such matters, will find in it information which will be of service to them.

A trade mark must be a "distinctive" sign, and not merely a descriptive adjective or word, as no person has the right to appropriate to himself any word or sign which is the common property of the public.

The use of a trade mark is to afford a ready means by which the public may recognise any particular goods, and hence its value.

A trade mark must comply with one or more of five definition clauses which are fully explained, but there are also several restrictions to be specially noted. The Royal Arms cannot be registered, nor portraits of the King and Queen, or any member of the Royal Family; no word will be accepted in the possessive case, nor can letters of the alphabet or figures be registered,

In the official classification list, goods are divided into fifty classes for which a trade mark can be registered, and separate registrations are required for each class. Some of these classifications are rather quaint; for instance, Class 14, goods of precious metals (including aluminium, nickel, Britannia metal).

Besides those of Great Britain, this book deals with foreign and colonial trade marks, though only in a general way.

The book is clearly written and well printed, and indexed.

The Chemistry of Organic Colouring Matters ("Chimie des Matières Colorantes Organiques"). By R. Nietzki, Professor at the University of Bâle. Translated into French from the Third German Edition, and Revised from the Fourth German Edition by C. Vaucher, C. Favre, and A. Guyot. Paris: G. Carré and C. Naud. 1901. Pp. 447.

Though there are some excellent books in France dealing with colouring matters, especially those by M. Lefèvre and MM. Seyewetz and Sisley, they are all too bulky, and their study would require a large amount of time. The present volume has the double merit of being both concise and complete; it is not intended solely for the use of those who wish, later on, to make colouring matters their principal study, but also for the more numerous class who are daily engaged in their application.

Dyers, paper-makers, lacquer makers, and in fact all those who have to do with the manufacture and dressing of skins, leather, feathers, waxes, &c., see the increasing necessity of no longer using products whose chemical nature is unknown to them; it is evident that this knowledge must be of great assistance, and will prevent many useless experiments.

The division of colouring matters into natural families was first inaugurated by M. Nietzki in the year 1886. The best proof of its excellence is that it has been adopted universally, both in literature and in teaching.

The book is divided into thirteen different sections or chapters, beginning with nitrated colouring matters, after which the author goes through all the other groups and families, such as the oxyquinones, the di- and tri-phenylmethanes, quinoneimide and its derivatives, aniline black, quinoleine and acridine, the oxyketones, xanthones, flavones, and coumarines, &c., winding up with a chapter on colouring materials of unknown composition.

The index is exceedingly complete, and is divided into two parts, one alphabetical and the other bibliographical. There is also a small list of errata, none of which, however, is of great importance, being principally slight mistakes in spelling.

CORRESPONDENCE.

PETROLEUM LAMPS.

To the Editor of the Chemical News.

Sir,—I have just obtained a copy of Captain Thomson and Boverton Redwood's "Handbook of Petroleum," and find, on page 272, Appendix XI., suggestions for the care and use of petroleum lamps. As I have had considerable experience in shale oils here, I would like to add to them.

In all mineral oil lamps the oil continually creeps all over the reservoir, especially when the metal work is fastened on with plaster-of-Paris and size. This can be completely prevented by smearing the joint with glycerin, which can be easily done by dipping the forefinger in it and going once or twice round the joint inside the lamp. It is necessary to do this before oil is used; that is, when the lamp is new, as the glycerin will not penetrate if the plaster is saturated with oil, and conversely, the oil will not penetrate if it is saturated with glycerin. It is advantageous to lubricate all tap-plugs, washers, &c., with glycerin when they are brought in contact with mineral oil of any kind.

Under No. 14 it is said:—"Do not continue to burn the oil until it is completely exhausted. It is best to keep the lamp well filled." If this is done, the oil in glass reservoirs gradually becomes yellow, especially if exposed to light, and such oil encrusts the wick and burns badly. Lamps in churches are used seldom, and hence the oil is always in that condition. Lamps should be emptied occasionally, and the residues may be collected and burned off in one filling to a small bulk, the residue being thrown away.

Glass lamps when not in regular use should be kept in the dark.—I am, &c.,

W. A. Dixon.

97, Pitt Street, Sydney, February 17, 1902.

Researches on Calcium Silicide, $CaSi_2$.—H. Moissan and W. Dilthey.—Lime, when kept in a state of fusion in presence of an excess of silicon, causes the formation of a compound of formula $CaSi_2$, analogous to Vöhler's silicide. The action of this new silicide on water cannot be compared with the action of calcium carbide. Water is very slowly decomposed by this silicide, with evolution of hydrogen. Dilute hydrochloric acid attacks it much more rapidly; hydrogen is evolved, but no solid silicon hydride is produced.—*Comptes Rendus*, cxxxiv., No. 9.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees o temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxiv., No. 9, March 3, 1902.

New Synthesis of Methane.—Paul Sabatier and J. B. Senderens.—The authors compare the various syntheses of methane which have been effected up to the present, and add to these their own method of direct hydrogenation, by the action of gaseous hydrogen in presence of recently reduced nickel, either on carbon dioxide or on carbon monoxide.

Dilatation of Steel at High Temperatures.—Georges Charpy and Louis Grenet.—From a series of tables the authors have drawn up by means of direct measurement, it is seen that the dilatation of steel at low temperatures presents curious variations according to the amount of nickel present in the steel; but the coefficients of dilatation increase rapidly with the temperature so as to mask these variations.

Specific Heat and Atomic Mass of Vanadium.—C. Matignon and E. Monnet.—The authors (1) prove the existence of the crystalline compound AlV_3; (2) find certain interesting properties of ferro-vanadium; (3) determine the specific heat of the metal vanadium to be 0·1565 between 15° and 100°; (4) show that the atomic mass, defined from Dulong and Petit's law, agrees with the atomic mass defined from considerations of isomorphism; (5) establish a simple method of estimation of vanadium.

Some Thallic Compounds.—V. Thomas.—The author believes that many of the thallic compounds hitherto accepted as definite are really mixtures of isomorphous compounds. This he proves to a certain extent.

Dioxy-tariric and Keto-tariric Acids.—M. Arnaud.—The oxidation of tariric acid—

$$CH_3—(CH_2)_{10}—C\equiv C—(CH_2)_4—CO_2H,$$

either by MnO_4K or by nitric acid, gives a dioxy-tariric acid, $C_{18}H_{32}O_4$. In this case tariric acid behaves as stearolic acid. The best method of causing the oxidation consists in the use of fuming nitric acid and stopping the rapid action by sudden cooling. After washing the crude product with water, the dioxy-tariric acid can be purified by crystallisation from concentrated alcohol. The yields are satisfactory. The acid is in the form of thin brilliant plates of a pale yellow colour, melting at 98°. Analysis proves the formula to be $C_{18}H_{32}O_4$.

Products of Condensation of Tetramethyldiamidobenzhydrol with some Primary Aromatic Amines with the Para-position occupied.—A. Guyot and M. Granderye.—The authors prepare, by condensing the primary aromatic amines having the para-position occupied with tetramethyldiamidobenzhydrol in dilute hydrochloric acid, not the products which theory would seem to suggest, but bodies of abnormal condensation—leucoauramine and azomethinic compounds.

New Reactions of Organo-metallic Derivatives.—E. E. Blaise.—By the use of organo-zincic compounds the author was enabled to prepare the primary β-oxy acids, mono- or di-substituted in α, oxy-acids of which scarcely any representatives are known, and hydracrylic acid. This method also allows of the preparation by simple dehydration of the non-saturated acids which correspond to the mono-substituted α-oxy-acids.

Compounds of Alcohol with the Chlorides of Manganese and Cobalt.—F. Bourion.—The author obtains two new compounds of cobalt and manganese with alcohol, and determines several physical properties of these bodies. He also finds a means of separating cobalt and nickel by the solubility of their anhydrous chlorides in alcohol.

Bulletin de la Société Chimique de Paris. Series 3, Vol. xxv., No. 14.

A Sample of Crystallised Lime.—A. Jouve.—Already noticed.

Condensation of Cetipic Ether with the Orthodiamines.—R. Thomas-Mamert and A. Striebel.—The authors give the results obtained by the condensation of cetipic ether with orthophenylenediamine and its derivatives and some tetramines. They include benzoquinoxaline-diethyloate of ethyl; the action of ethylate of sodium; the constitution of benzo-β-ketopentamethyleneazinemethyloate of ethyl; the action of carbonate of sodium in solution on benzo-β-ketopentamethylene-azinemethyloate of ethyl; benzo-β-ketopentamethylene-azinemethyloic acid; and many other bodies.

The Nitrated Bases in Roumanian Petroleum.—Dr. A. B. Griffiths and N. J. Bluman.—Roumanian petroleum is composed of hydrocarbides of various series, such as methane, ethylene, acetylene, and benzene, as well as cyclohexanes or saturated cyclic hydrocarbides, (C_nH_{2n}). The authors have recently analysed a sample of raw Roumanian petroleum of $D_{15} = 0·8445$, and they found the following products:—

	Per cent.	
Benzine	10·65	
Illuminating oil (1st quality) ..	61·20	81·28
,, ,, (2nd quality) ..	20·08	
Paraffin..	2·83	
Coke and loss	5·24	

These figures justify the praise which has been bestowed on Roumanian petroleum, as a raw petroleum yielding 81 per cent of illuminating oil should be the basis of an important industry. The authors agree that this petroleum contains very little oxygen (less than 1 per cent), and they have recently discovered a basic nitrated compound in it having the odour of pyridine. This was effected by shaking up the petroleum with a 24·5 per cent aqueous solution of sulphuric acid. From this mixture, after adding an alkali and extracting with ether, they obtained a deep brown coloured, thick, oily liquid, boiling at 117°. This substance is very slightly soluble in cold water (pyridine is easily soluble); it is, however, soluble in ether, alcohol, benzene, and the mineral acids. It gives a crystalline precipitate with chloride of platinum and with ferrocyanide of potassium. The analysis of this body when purified gives the following results:—

	I.	II.	III.
Carbon	75·3	75·0	75·8
Hydrogen.. ..	6·8	6 6	6·5
Nitrogen	17·9	—	17·7

By treating a warm alcoholic solution of this substance with metallic sodium they obtained piperidine ($C_5H_{11}N$). This proves that the substance is pyridine (C_5H_5N) or a substance analogous to it.

A Biochemical Differentiation of the two principal Ferments in Vinegar.—G. Bertrand and R. Sazerac.—The authors have observed a very distinct differential character between two species of acetic ferments they often meet with, viz., the bacteria of vinegar (*Mycoderma aceti*) and the bacteria of sorbose (*Bacterium xylinum*). The first species is of the type of a ferment. Two samples of this, from different sources, have been examined, and they differ very slightly one from the other. Conical vessels containing 50 c.c. of broth, with 0·5 per cent of yeast and about 2·5 c.c. of alcohol, were infected and kept at a temperature of 28°. The acetic acid found was as follows:—

	Six days. Grms.	Ten days. Grms.	Seventeen days. Grms.
Microbe from Orleans	1·53	2·81	2·45
Microbe from Paris ..	1·43	2·35	2·52

The second species, from sorbose, was sown in a broth containing glycerin and peptone, and after two or three days a liquor was obtained precipitating Fehling's solution in the space of a few seconds; it oxidises the glycerin and forms dioxyacetone or propanediolone.

The Sensitiveness of the Methods for Detecting Salicylic Acid in Wines.—A. J. Ferreira da Silva.—After discussing various methods for detecting salicylic acid in wines, the author comes to the conclusion that the official German method is the best. The results obtained by four different methods are set out in a table.

Method of Preparation of the Lower Brewery Yeasts having the Property of Fermenting at a High Temperature, and How to Use them.—Georges Jacquemin.—Up to the present the methods of making beer could be divided into two great classes, according to whether the yeast was "high" or "low." The low yeasts are always used at a temperature below 10°, while the high yeasts are always used at a temperature above 10°. The beers obtained by the low fermentation have always a tendency to re-ferment if kept at a higher temperature than that of the original fermentation. The author has succeeded in modifying the conditions of existence of all the low fermentation yeasts, making them acquire the property of fermenting at a high temperature, even above 20°, while retaining in the beer the characteristic qualities demanded by the public.

MISCELLANEOUS.

Dionine.—This is simply a fancy name for ethylmorphine hydrochloride, which was first prepared many years ago by the French chemist Grimaux. The alkaloid and the salt are regularly prepared by British manufacturers, and sold under their proper systematic names. Ethylmorphine is a homologue of codeine (methyl morphine), and has been shown by Professor Stockmar and others to have the same physiological action.

Directory of Paper-Makers.—We have received a copy of the above Directory for 1902, published by Marchant, Singer, and Co. The lists of paper-makers of the United Kingdom, under all the various headings, have been carefully and authentically revised to date. Additions have been made to the list of trade designations used as water-marks, &c., in papers, stationery, &c., bringing the total to over 4000; this feature will be of very great value to printers and stationers. There is also a complete classification of advertisers, forming a directory of paper-makers' principal engineers, purveyors of raw materials, paper mill suppliers, &c.; the whole making a useful book of reference for the trade.

On Pilocarpine.—A. Pinner and E. Kohlhammer.—The permanganic oxidation of pilocarpine eliminates the whole of the nitrogen from this base in the form of ammonia and methylamine, and gives a bibasic acid, $C_8H_{14}O_6$, of which the salts lose one molecule of water by the action of heat, and then correspond to a bibasic acid, $C_8H_{12}O_5$. $C_8H_{14}O_6$ is pulvic acid. Peroxide of hydrogen reacts on pilocarpine in the same way as permanganate. The sulphochromic mixture only reacts at the temperature of the water-bath, and gives pilocarpeic acid, $C_{11}H_{16}N_2O_5$. According to the authors, pulvic acid would be identical with the acid $C_7H_{10}O_4$ of Jowett (*Proc. Chem. Soc.*, vol. xvi., p. 124), and with the pyridinotartronic acid of Hardy and Calmels. Pulvic acid gives a deliquescent salt of potassium, $C_8H_{12}O_6R_2$ ($C_8H_{10}O_5K_2$ at 100°):—$C_8H_{12}O_6Ag_2$, $C_8H_{12}O_6Ba$ loses $\frac{1}{2}H_2O$ at 180°; —$C_8H_{10}O_5(C_2H_5)_2$ boils at 181—183° under 26 m.m. pressure, and 293° at 755 m.m. pressure. Pilocarpeic acid, $C_{11}H_{16}N_2O_5\,H_2O$, is gummy, and fuses at <100°.—*Berichte*, vol. xxxiii., p. 2357.

Action of Metallic Sodium on the Bromised Ethers.—B. N. Menschoutkine.—The author has examined the action of metallic sodium on the ethers $CH_2Br.CH_2.O.C_2H_5$ and $CH_2Br.CH_2.CH_2O.C_2H_5$, prepared by the method he devised in collaboration with M. Volhof, and which consists of reacting with ZnBr, ZnO, or even CaO on alcoholic solutions of the dibromides. These bromised ethers are liquids of an agreeable odour; bromethylic ether boils at 127—128° d_0° = 1·3956, for bromopropylic ether, boiling at 149—150° d_0° = 1·2697. The reaction of metallic sodium on these bodies in etherial solution commences without the application of heat; an abundant evolution of gas takes place, and the temperature rises; this action ceases after about twenty-four hours. The mixture, after standing for several days at the ordinary temperature, is heated on the water-bath, and the gases given off were recognised as being ethylene or propylene, according to which ether was used. The liquid products formed are principally ethylic ethers of the ethylenic and propylenic glycols, but there are also portions having a higher boiling-point which appear to contain the ethylic ethers of the tetramethylenic and hexamethylenic glycols. The principal reaction can be expressed (for bromethylic ether) by the equation:—

$$2CH_2Br.CH_2.O.C_2H_5 + 2Na = = C_2H_4 + C_2H_5.O.CH_2.CH_2.O.C_2H_5 + 2NaBr.$$

—*Journ. Soc. Phys. Chim. R.*, vol. xxxii., p. 355.

The Separation of Aluminium and Glucinum by means of Hydrochloric Acid.—F. S. Havens.—The author, in collaboration with Mr. Gooch, has shown that hydrated chloride of aluminium is insoluble in ether saturated with hydrochloric acid, and that Fe_2Cl_6 is not; this allows of the separation of the two metals. Aluminium can be separated from glucinum in a similar way; the two metals being brought to the state of chlorides, half the volume of ether is added to the aqueous solution; this is then saturated with hydrochloric acid gas, and half the volume of ether again added, and the mixture again saturated with hydrochloric acid. In this manner a crystalline precipitate of $Al_2Cl_6 + 12H_2O$ is obtained, which is collected on an asbestos filter, and washed with ether saturated with hydrochloric acid gas. This precipitate is dried at 150°, then calcined progressively under a layer of pure mercuric oxide; the alumina is finally weighed. This latter is free from glucinum; as for the glucinum, it is found entirely in the filtered etherial liquor. After having driven off the whole of the ether and the free hydrochloric acid by evaporation, the glucina can be estimated, either by precipitating with NH_3 or by evaporating the solution containing the $GlCl_2$ with nitric acid, and then calcining the nitrate formed.—*Zeit. Anorg. Ch.*, vol. xvi., p. 15.

NOTES AND QUERIES.

Note on the Cleansing of Mercury.—The necessity of having clean mercury for an experiment led me to try the methods suggested in "Thorpe's Dictionary of Chemistry." These are—(*a*) The use of sugar, (*b*) very dilute nitric acid, (*c*) sulphuric acid as digesting agents. The first method was not only bothersome, but a failure. Even dilute nitric acid attacks the metal after a time. Sulphuric acid, however, proved quite successful. Digestion with strong sulphuric acid (1 : 8) is an excellent method of cleansing mercury, and after three or four digestions with it, dilutions, and filtrations, even the dirtiest mercury is cleansed.—FRANCIS GEORGE COUSINS, Golding Grammar School, Kent.

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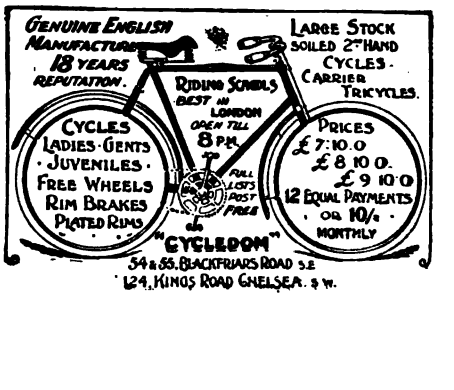

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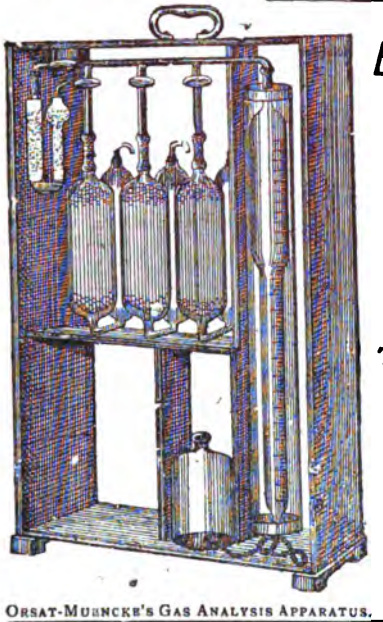

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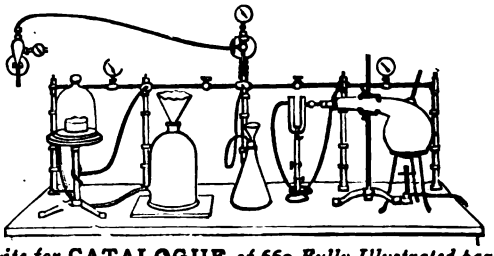

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VOL. LXXXV., No. 2210.

THE EFFECT OF LIME ON THE INSOLUBLE PHOSPHATES IN SOILS.

By WALTER F. SUTHERST, Ph.D., A.I.C.

The importance of adding lime to land and its many advantages are, of course, well known to every farmer, but one of its effects seems to be overlooked, viz., its action on those phosphates which are generally considered valueless as plant foods, *e.g.*, those of iron and aluminium. These occur in nearly every soil, and are of two sources: one being from the disintegration of rocks containing vivianite (ferrous phosphate) and wavellite (aluminium phosphate), the other source being a result of the reversion of superphosphate of lime added to soils containing oxides of iron and aluminium.

Phosphates of calcium (that is, the tri- and di-), though insoluble in water, are, nevertheless, not valueless as plant foods, since the weakest acids, such as carbonic and the acids in the roots of plants, are capable of dissolving them, and thus rendering them capable of absorption. Since the phosphates of iron and aluminium are insoluble in carbonic, and very sparingly soluble in organic acids, their food value is comparatively small, and a long time is needed before they could be absorbed by the root saps.

The method usually employed in England for determining the value of neutral phosphates is that recommended by Dr. Bernard Dyer, and consists in treating the phosphate in question with a 1 per cent solution of citric acid (this strength being the nearest to the acidity of the root saps); and this method I have used in the determinations carried out.

To show the relatively low value of the iron and aluminium phosphates as such, the solubilities of these were first determined as follows:—

One grm. each of ferrous, ferric, and aluminium phosphates were placed in a small flask containing 100 c.c. distilled water, and to each was added 1 grm. citric acid, then allowed to stand twenty-four hours with occasional shaking. At the end of that time the liquid was filtered off, the residue well washed with distilled water, and in half the filtrate (= 0·5 grm. substance) the soluble phosphoric acid was determined by adding ammonium molybdate and nitric acid and heating on the water-bath three hours. After cooling, the clear supernatant liquid was filtered off and the residue washed four times by decantation with 2½ per cent nitric acid. The precipitate was then dissolved in ammonia and to this magnesia mixture added, and after standing three hours the ammonium-magnesium phosphate filtered off, washed with 2½ per cent ammonia, the filter and contents placed in a platinum crucible, and ignited first over the Bunsen flame and finally over the blowpipe.

0·5 grm. $Fe_3(PO_4)_2$ gave 0·0388 grm. $Mg_2P_2O_7$, equal to 2·475 per cent P_2O_5 dissolved.
0·5 grm. $Fe(PO_4)_2$ gave 0·0432 grm. $Mg_2P_2O_7$, equal to 2·755 per cent P_2O_5 dissolved.
0·5 grm. $Al(PO_4)$ gave 0·0502 grm. $Mg_2P_2O_7$, equal to 3·203 per cent P_2O_5 dissolved.

The three phosphates on analysis contained as total phosphoric acid:—(1) Ferrous phosphate, 25·924 per cent P_2O_5; (2) ferric phosphate, 23·255 per cent; (3) aluminium phosphate, 28·680 per cent. The proportion of citric acid-soluble phosphoric acid to total would be—

$Fe_3(PO_4)_2$	10·64
$FePO_4$	10·62
$AlPO_4$	11·16

When slaked lime is allowed to act on the above phosphates in the presence of water, the greater part of the phosphoric is rendered soluble in citric acid, especially in the case of the ferric phosphate (the one most commonly found in the soil), the lime evidently combining with the phosphoric acid, setting free hydrates of iron and aluminium; the change of the light yellow ferric phosphate to a reddish brown precipitate proving this decidedly.

The action of lime was carried out experimentally as follows:—One grm. of the phosphate was placed in a small flask and mixed with 100 c.c. distilled water, then 2 grms. pure slaked lime added; the contents of each flask then well agitated and allowed to stand for periods of twenty-four, forty-eight, and seventy-two hours with constant shaking. At the end of twenty-four hours 1 grm. of citric acid was added to three of the different phosphates, and the calculated amount of citric acid necessary to neutralise the excess of lime present, and after standing twenty-four hours filtered off, and the dissolved phosphoric acid estimated as above. The following results were obtained:—

Time. Hours.	$Mg_2P_2O_7$. Grms.	Equal to P_2O_5.	Proportion of soluble to total P_2O_5.
	I. *Ferrous Phosphate.*		
24	0·1532	19·55	75·42
48	0·1736	22·15	85·45
72	0·1746	22·26	85·88
	II. *Ferric Phosphate.*		
24	0·1721	21·96	94·45
48	0·1756	22·41	96·38
72	0·1760	22·45	96·55
	III. *Aluminium Phosphate.*		
24	0·1441	18·45	64·33
48	0·1558	19·88	69·31
72	0·1564	20·65	72·00

In the same manner experiments were tried using calcium carbonate instead of slaked lime, but in no case, even at the end of fourteen days, was there any increase in the solubility.

From the above results it will be seen that it is essential that the lime should be in the form of hydrate, the carbonate being of no value whatever. Since lime is often applied to land in a very careless manner, it naturally follows that the beneficial effects of it are often lost. If left exposed for a long time to the air, it changes slowly to carbonate, due to absorption of carbonic acid from the air and rain, and much is lost in this way when the lime is air-slaked. Too much stress cannot be laid on this matter, and all farmers should take care to spread the lime as soon as it has crumbled to powder.

The Cheshire Agricultural College,
Holmes Chapel.

On Certain Regular Relations Observed in the Distillation of Dilute Aqueous Solutions of Phenol.—Al. Neumann and W. Muller—Five hundred c.c. of an aqueous solution of phenol of known strength are boiled for thirty minutes, and care is taken to keep the volume constant by adding water by means of a funnel fitted with a tap; the phenol contained in 100 c.c. of the liquid which passed over during this thirty minutes distillation is then estimated. By repeating this experiment on solutions of different strengths, the authors found that 100 c.c. of the distillate constantly contained three times less phenol than the 500 c.c. of solution at the commencement of the distillation, and they calculate from this that at 100°, near the surface of the boiling solution, the proportion of phenol in the vapour should always be double the amount of that in the liquid at equal volumes.—*Berichte*, vol. xxxiv., p. 224.

THE OXIDATION OF NITRITE BY PERMANGANATE.

By JOHN WADDELL, B.Sc. (Lond.).

In the titration of a nitrite by potassium permanganate it is usual to add a large excess of sulphuric acid. The oxidation may therefore be considered as an oxidation of nitrous acid to nitric acid.

It happened to be a matter of interest to me to learn whether excess of acid is necessary, or whether a nitrite can be oxidised to a nitrate. As I found nothing on this point in the literature of the subject I carried out the following little investigation with sodium nitrite:—

I started out on the assumption that the reaction between sodium nitrite and potassium permanganate in acid solution is represented by the equation—

$$5NaNO_2+9H_2SO_4+2KMnO_4= =5NaHSO_4+2KHSO_4+2MnSO_4+5HNO_3+3H_2O,$$

and weighed out the reagents accordingly, taking roughly 10 grms. of sodium nitrite, 26 grms. of sulphuric acid, and 9 grms. of potassium permanganate, each of these quantities being dissolved in water, and the solutions being made up to a litre.

The permanganate was standardised against Mohr's salt; 4 grms. of Mohr's salt required 36·3 c.c. of the permanganate solution, which therefore contained 8·883 grms. solid permanganate per litre. The nitrite was then titrated against the permanganate in two ways. In the first titration 3 c.c. or 4 c.c. of sulphuric acid was added to water, and the solution cooled; then about 25·5 c.c. of permanganate solution added, and the whole made up to approximately 100 c.c. This acidulated permanganate was added to 24·9 c.c. of nitrite also diluted to 100 c.c. From previous trials these quantities were known to be approximately equivalent. The permanganate was completely decolorised, and more was added from the burette until a pink colour was obtained which lasted for ten minutes. The total amount of permanganate was 26·2 c.c.

Another titration was made in accordance with Lunge's method for estimating nitrous fumes dissolved in sulphuric acid. About 7 c.c. of strong sulphuric was added to about 100 c.c. of water, the temperature becoming 40° C.; 20 c.c. of permanganate was added to this, and then nitrite was run in. The decolorising took a few seconds, and I found I had very slightly overshot the mark, and needed to add 0·25 c.c. permanganate in order to bring back the pink colour. In this test 20·25 c.c. permanganate were decolorised by 19·30 c.c. nitrite.

The two titrations gave:—

1 c.c. nitrite = 1·0522 c.c. permanganate
1 „ „ = 1·0493 „ „

Mean .. 1·0507

and the solution of nitrite contained 10·190 grms. sodium nitrite per litre.

The sulphuric acid was estimated by precipitating with barium chloride, and proved to contain 25·74 grms. H_2SO_4 per litre.

A few preliminary experiments showed that the sulphuric acid necessary was much less than required by the equation at first assumed. The most satisfactory method of procedure was found to be to add the mixture of acid and permanganate to the nitrite; when the nitrite was added in the cold to the permanganate a muddy precipitate of manganese hydroxide was formed which was difficult to get rid of. At 40° C. there was not so much precipitate on adding the nitrite to the permanganate, and the result of one determination in this way is given.

To 26·15 c.c. permanganate 8·75 c.c. sulphuric acid was added, and water to make up to about 100 c.c., and the temperature was raised to 40° C. When nitrite was run in, the liquid became muddy, but on the addition of 24·80 c.c. of nitrite the precipitate soon settled, leaving the supernatant liquid colourless. There was considerable deposit of hydroxide, which became less, but did not entirely disappear at 60° C. The liquid was practically neutral, but became acid on addition of 0·2 c.c. acid, and after a few hours the residue almost entirely disappeared.

Though according to Lunge, when excess of acid is present, it is better to add the nitrite to the acidified permanganate, for my purpose the converse process was preferable, and my final experiment was the following:—

8·75 c.c. of sulphuric and 25·88 of permanganate were added to 100 c.c. of water at 40° C., and a little of this mixture added to 25 c.c. of the nitrite. A muddiness was produced, and a few drops more of acid were added, making in all 8·9 c.c. The nitrite was slightly warmed, and then all of the permanganate mixture was added. The liquid became clear and colourless. A little more permanganate was added from the burette, no additional acid being put in. The colour took some minutes to disappear. The last addition was 0·2 c.c., and the colour did not disappear after ten minutes. There was a very slight cloudiness; the liquid was neutral. As the last addition of permanganate seemed to be a slight excess, I assumed that 0·1 c.c. would have been enough to add. This would have made a total of 26·18 c.c. of permanganate for 25 c.c. of nitrite, or 1 c.c. nitrite = 1·0472 c.c. permanganate; a result not far from that obtained with a large excess of acid.

The actual amounts by weight of the reagents was:—

0·2325 grm. permanganate
0·2547 grm. nitrite
0·2291 grm. sulphuric acid

This very nearly corresponds with the equation—

$$5NaNO_2+3H_2SO_4+2KMnO_4= =5NaNO_3+K_2SO_4+2MnSO_4+3H_2O,$$

according to which 0·2325 grm. of permanganate would require 0·2537 grm. of nitrite and 0·2162 grm. of sulphuric acid.

It appears, therefore, that it is not necessary to add more acid than just enough to make *neutral* sulphates and nitrates.

School of Mining, Kingston, Canada.

A NEW METHOD FOR THE ESTIMATION OF GOLD AND SILVER IN PYRITES.

By W. BUDDËUS.

The usual methods are scorifying with lead in small dishes in a muffle, or fusing with lead and a flux, then cupelling in both cases. The first of these methods is the most exact, but it is expensive on account of the large amount of material used and the time required. Even very satisfactory results are too low, however, up to a certain point, because of the volatilisation due to the high temperature of the muffle, and also because some of the precious metal remains fixed in the regulus which is always formed between the lead and the slag.

In this new method the sample is ground sufficiently fine to pass through a sieve having 300 or 400 meshes per square centimetre; 100 grms. of an ore containing 50 grms. or more of gold per 1000 kilos., or 200 grms. for ores which contain less than 50 grms. per 1000 kilos., are placed in a crucible, preferably one of white Paris clay, of 70 or 130—140 c.c. capacity, according to the weight of the sample taken.

The crucible is lightly covered and placed in a muffle heated to redness, for half to three-quarters of an hour, until all trace of the sulphur flame disappears. It is placed on a sheet of iron to cool, and the contents then emptied into a glass vessel or porcelain dish of 1 or 2 litres capacity.

The roasted ore, which is principally sulphide of iron, is only loosely aggregated, and can be easily detached from the crucible, and the dust brushed out with a camel-hair brush.

It may be stated, generally, that the mass has taken the form of the interior surface of the crucible, but it need not be broken up. It is then treated with a mixture (250 or 500 c.c.) of hydrochloric acid free from arsenic, with its own volume of water. After standing for about an hour in a warm place, another 250 or 500 c.c. of the acid mixture are added, and the whole boiled for some time. The sulphide of iron, which formed eight-ninths of the whole mass, is dissolved rapidly, leaving behind the whole of the gold and silver, with the silica and clay of the gangue.

When the sulphide is completely decomposed, the solution is diluted with water until the crucible or dish is nearly full; it is then filtered.

The residue is washed two or three times with water, then dried, transferred, with the filter-paper, to a Hessian crucible of 150 or 300 c.c. capacity, mixed by means of a spatula with 50 or 100 grms. of assay lead and 5 or 10 grms. of borax, and finally fused in the muffle. It is of advantage to use 100 or 200 grms. of a mixture of equal parts of acetate of lead and caustic soda, instead of assay lead.

After cooling, the crucible is broken, the lead is detached and cupelled immediately; the gold and silver are then parted in the usual manner.

The cost of an assay of 200 grms. of ore, according to the old method, which necessitates the use of 1400 grms. of pure lead at 1 mark 20 pfennigs per kilo., besides the scorifying dishes and cupels, and allowing 1 mark for heating, &c., is 1 mark 88 pfennigs; while, by the new method, which only requires 500 c.c. of hydrochloric acid, 100 grms. of assay lead, and a crucible, the cost, including the heating, &c., varies from 37 pfennigs to 72 pfennigs.

The results are perfectly accurate and concordant among themselves; check assays never differ by more than 1 m.grm.

The results, as proved by comparative assays, are uniformly higher than those obtained by the old method.—*Chemiker Zeitung*, xxiv., p. 922.

INTERNATIONAL CONGRESS OF APPLIED CHEMISTRY.

We wish to draw the attention of our readers to the fact that the Fifth International Congress of Applied Chemistry will take place at Berlin during the Whitsuntide Holidays in 1903, under the Presidency of Dr. Clemens Winkler. The House of the Imperial Parliament (Reichstag) has been placed at the disposal of the Congress, and Geheimrath Professor Otto N. Witt has been nominated President of the Organising Committee; Dr. Bottinger, Member of the German Parliament, acting as Treasurer. A fund of about £3000 has already been collected by voluntary subscriptions from societies and private individuals towards the expenses of the Congress.

The object of these Congresses is to further the interests of "Applied Chemistry" in the widest sense of the words. They embrace both Industrial and Analytical Chemistry; in the latter department one of the chief objects is to enable chemists to decide on standard methods for the testing and assaying all kinds of chemical products and raw materials, and to bring about international agreements for the employment of the selected processes. Much useful work has already been effected in this direction at the former Congresses held in Paris, Brussels, Vienna, and again in Paris.

Great interest has been taken in these meetings by different Continental Governments, and an official representation may be expected on the part of some of them on the forthcoming occasion.

The absence of many English chemists on previous occasions has been much remarked, although this probably arose from the Congresses not being better known. It would seem to be greatly to the advantage of English science that this nation should be well represented at Berlin. Probably the best commencement would be for some of the leading chemists to form a committee for carrying out the preparatory work needed to enable this country to take its rightful place among the nations who will be represented at Berlin next year. We should think that the selection of such a committee would fall well within the scope of the British Association, and it is to be hoped that under the auspices of so eminent a chemist as Professor Dewar, the President, a representative committee with this object will enter into existence during the Belfast Meeting.

ON THE FUSION OF QUARTZ IN THE ELECTRIC FURNACE.*

By R. S. HUTTON, M.Sc.

During recent years a good deal of attention has been paid to the working of fused quartz with a view to its use in the construction of apparatus. (*Vide* C. V. Boys, *Phil. Mag.*, 1887; H. L. Callendar, *Journ. Iron and Steel Inst.*, 1892, i., 165; W. Crookes, "On the Spectra of Argon," *Phil. Trans.*, 1895, 186 (A), 245; R. Threlfall, "Laboratory Arts," 1898, esp. p. 199; A. Dufour, *Comptes Rendus*, 1900, cxxx., 775; A. Gautier, *Comptes Rendus*, 1900, cxxx., 816; W. A. Shenstone, *Nature*, 1900, lxi., 540; W. A. Shenstone and H. G. Lacell, *Nature*, 1900, lxii., 20; W. A. Shenstone, *Proc. Roy. Inst.*, 1901; *Nature*, 1901, lxiv., 65 and 126).

Owing to the exceedingly small coefficient of expansion of this material (H. Le Chatelier, *Comptes Rendus*, 1889, cviii., 1046, and 1900, cxxx., 1703; H. L. Callendar, *vide* Shenstone, *Nature*, 1901, lxiv., 66), and to its high melting-point, it would doubtless find many applications for making vessels in which to study the properties of substances at high temperatures, and especially for making high temperature gas thermometers. Further, since fused silica withstands very sudden changes of temperature without showing any tendency to crack, probably many industrial uses could be found for it as soon as a more economical method of fusing and working it is forthcoming. Up to the present, the oxyhydrogen blowpipe has been used almost exclusively, and, in the hands of Shenstone and Lacell and of Dufour, has led to very satisfactory and promising results. The process is, however, expensive, and is also very slow, since the oxyhydrogen flame is at a temperature only slightly above the fusing-point of silica, and, in fact, it is only at a certain point in the flame that the fusion can be carried out at all.

Moissan ("Le Four Electrique," 1897, p. 49; A. Gautier, *Comptes Rendus*, 1900, cxxx., 816) has studied the effect of the electric arc upon silica, and has noted that it fuses, and is easily volatilised; but he studied more particularly the reduction products, carborundum and silicon, which remain behind when silica is heated for some time in an electric furnace, and, according to Gautier, did not succeed in making apparatus of this material. Upon repeating Moissan's experiment on the volatilisation of flint stones as a lecture experiment with an electric-arc furnace using 300 ampères at 50 volts, I was struck by the appearance of the residual unvolatilised silica, which was very glassy and in parts quite transparent. Experiments soon showed that, provided a small current of air is allowed to pass through the furnace, no reduction of the solid or liquid silica takes place, and, consequently, the arc can be used as a source of heat for fusing silica without fear of reduction spoiling the results.

* Read before the Manchester Literary and Philosophical Society, January 7th, 1902.

At first apparatus was fitted up for obtaining an arc-flame from inclined carbons by magnetic deflection downwards; in this way the arc-flame was made use of much in the same manner as the oxyhydrogen flame, and rods could be made easily by the method recommended by Shenstone. It was soon found, however, that a small arc using 15—20 ampères was not sufficiently powerful to ensure rapid work, and after working with a similar open arc, using about 50 ampères with separate circuit for the magnet, the furnace was again brought into use for fusing the quartz.

Whilst employing the open arc, however, several interesting observations were made, which may be worth while recording.

In the first place, supports of carbon can be used with advantage for bringing the quartz into the flame, but it is necessary that the carbon used for these supports, as also the carbons used for the arc, be as pure as possible, or else the quartz may be contaminated by the ash which falls upon it; for the supports I have throughout made use of graphitic carbon prepared in the electric furnace, which is very pure and easy to work; it can, moreover, now be obtained on the market at a reasonable price.

In building up small rods, grooves were cut in a plate of this carbon, and small pieces of prepared quartz were placed in these grooves, the quartz being melted together gradually from one end to the other of the grooves, by sliding the carbon plate gradually under the arc-flame. Small lens-shaped discs can be similarly prepared by placing pieces of the prepared quartz in a shallow carbon crucible, and, with a little practice, it is hoped that masses suitable for making lenses can be prepared in this way. The valuable optical properties of fused quartz are pointed out by Shenstone in his papers.*

When quartz is thus fused in the arc, it is quite easy to observe the reduction which takes place in the immediate neighbourhood of the arc, and causes a black stain on the surface, which can easily be made to disappear by bringing the heated mass for a short time away from the centre of the flame; in fact, the working of quartz in the arc-flame much resembles the working of lead glass in the ordinary blowpipe, except for the immense difference of temperature.

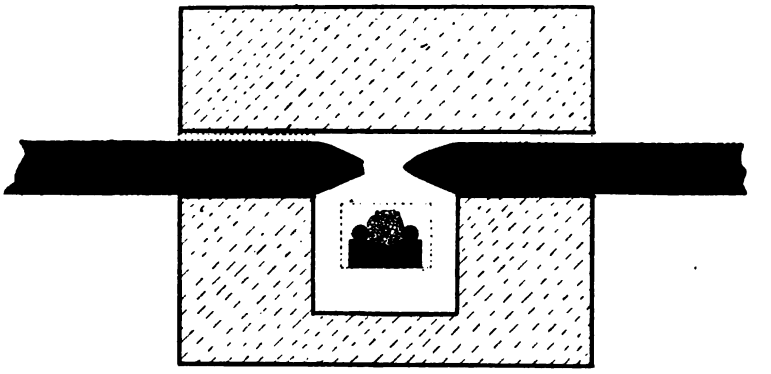
Owing to insufficient skill in the art of glass-blowing, and to the pressure of other work, I did not feel encouraged to proceed with these experiments with the open arc, but undertook a few experiments with the furnace which have led to promising results.

A Moissan arc furnace of the well-known type was modified by cutting passages in the sides, so that a carbon support charged with quartz could be passed under the arc and at right angles to it; a section of the furnace is seen in the accompanying figure.

* It should be mentioned that Messrs. Zeiss, of Jena, exhibited small lenses at the Paris Exhibition in 1900, made of "crystal de roche fondu," but the method by which the quartz was fused was not stated.

In this way one certainly makes more use of the heat of the arc, since the reflection from the cover prevents a great part of the heat being lost, and magnetic deflection of the arc, which in this case was not made use of, might be found a further advantage for economical working.

A current of about 300 ampères at 50 volts was usually employed, and with this power the quartz was seen to melt on the surface within a minute or so of commencement. Nearly all the experiments with this form of furnace were arranged with a view to preparing thick-walled tubes of quartz, with a core of about ½ inch. As seen from the figure, a rough mould was made of carbon with a carbon core, resting at each end on carbon supports. The mould was arranged usually for making tubes 10 to 12 inches long, but, obviously, any desired length of tube could be similarly constructed. The mould was filled with broken-up quartz of different degrees of fineness, but it was found that the best results were obtained when the quartz was not too finely powdered. The tubes obtained in this way can easily be separated from the carbon support and the core be withdrawn, since the silica does not adhere to the carbon.



Section of Electric Furnace. (One-fifth natural size).

Up to the present, I have not succeeded in preparing tubes quite free from bubbles, but the general appearance can be greatly improved by re-heating under the arc, preferably with mechanical rotation in order to get a more uniform result. Even in their present form, however, they would probably be of much use for constructing apparatus, as, owing to their thickness, there is plenty of material for blowing and drawing down purposes, and by this process the appearance of the material is, of course, much improved.

Pure white sand fused in a similar way always seems to give a much more opaque mass; but plastic quartz can take up a certain amount of sand without thereby impairing its transparency, and probably a small sand blast, blowing sand or fine quartz, could be used for thickening bulbs and tubes of quartz fused by this method.

It is to be hoped that those who have large power at their disposal will extend the application of this method, which may thus bring fused quartz apparatus within the reach of those wishing to make use of it in virtue of its interesting and valuable properties.

The experiments described in this paper were carried out in the Physical Laboratories at Owens College.

Research on Lactic Fermentation from observations of Electric Resistance.—MM. Lesage and Dongier.—Ostwald's apparatus, which allows of the measurement of electric resistance of liquids by Kohlrausch's method, can be utilised for the study of milk fermentation. It is found that the diminution in resistance is progressive, and the rapidity of the variation often changes, according to whether the milk is kept in closed or open vessels.—*Comptes Rendus*, cxxxiv., No. 10.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, March 19th, 1902.

Prof. J. EMERSON REYNOLDS, Sc.D., V.P.R.S., President, in the Chair.

MESSRS. Catchpole, Green, and Hanson were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. George H. A. Clowes, Gratwick Research Laboratory, Buffalo, U.S.A.; Henry Crookes, 109, Ladbroke Grove, W.; Walter Everitt, 83, The Grove, Ealing, W.; J. Arthur Gill, 19, Burma Road, Stoke Newington, N.; John T. Griffiths, Grammar School, Hanley Castle, Worcestershire; Sidney Isaac King, 49, Arundel Square, N.; Robert Mathieson, Rillbank, Innerleithen, Peebleshire; George Augustus Henry Mence, Ouse Villa, St. Ives, Hunts; Thomas Taylor, 12, Ancaster Drive, Great Western Road, W.; Frank Stanley Wood, 25, Suffolk Street, Newland, Hull.

The SECRETARY announced that the Treasurer, Professor TILDEN, had represented the Society at the recent celebration of the Jubilee of The Owens College, Manchester, and had presented the following congratulatory Address from the Society:—

"The President, Council, and Fellows of the Chemical Society desire on this the present occasion of the celebration of the Fiftieth Anniversary of the foundation of Owens College to express their high appreciation of the conspicuous services rendered to the cause of scientific education by the foundation and ever increasing activity of the College.

"They recall with especial satisfaction the contributions to Chemical Science which have emanated from the laboratories of Owens College under the direction of the successive eminent Professors of Chemistry who must be regarded as the immediate scientific successors of John Dalton, the founder of Modern Chemistry. With sincere wishes for the future development and extended usefulness of the College they desire to tender to the President, Council, Fellows, and Students an expression of hearty congratulation on the celebration of this their Jubilee.

J. EMERSON REYNOLDS, President.
WILLIAM A. TILDEN, Treasurer.
WYNDHAM R. DUNSTAN,
ALEXANDER SCOTT,
RAPHAEL MELDOLA,
} Secretaries."

Of the following papers, those marked * were read:—

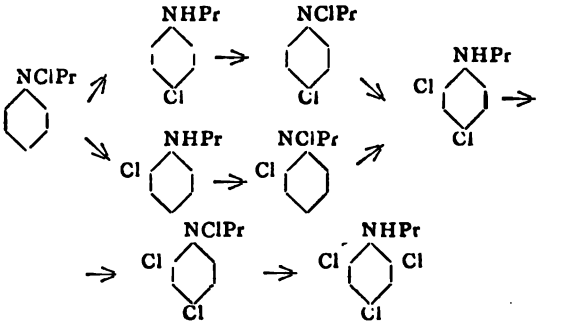
*43. "*Nitrogen Chlorides containing the Propionyl Group.*" By F. D. CHATTAWAY.

Aniline and the chloro-substituted anilines readily yield propionyl derivatives, which, on treatment with excess of hypochlorous acid, are converted into substituted nitrogen chlorides. In these reactions, hypochlorites are probably first formed, the nitrogen developing its higher valency, water being subsequently eliminated. The reaction is a reversible one:—

$$\begin{matrix} R \\ Pr \end{matrix}\!\!>\!NH + HOCl \rightleftharpoons \begin{matrix} R \\ Pr \end{matrix}\!\!>\!N\!\!<\!\!\begin{matrix} H \\ OH \\ Cl \end{matrix} \rightleftharpoons \begin{matrix} R \\ Pr \end{matrix}\!\!>\!N{\cdot}Cl + H_2O.$$

These nitrogen chlorides show all the characteristic reactions of the group, the chief feature of such reactions being the invariable replacement of the halogen by hydrogen.

When there is hydrogen, either in the para- or the ortho-position, transformation of the nitrogen chloride into the isomeric substituted anilide readily takes place according to the scheme:—



The following compounds have been prepared:—

Chloroiminopropionanilide, $C_6H_5{\cdot}NCl{\cdot}COC_2H_5$, colourless plates, m. p. 77°.

p-*Chloropropionanilide*, $C_6H_4Cl{\cdot}NH{\cdot}COC_2H_5$, plates, m.p. 141°.

Chlorimino-p-chloropropionanilide, $C_6H_4Cl{\cdot}NCl{\cdot}COC_2H_5$, colourless plates, m. p. 55°.

Bromoimino-p-chloropropionanilide, $C_6H_4Cl{\cdot}NBr{\cdot}COC_2H_5$ yellow prisms, m. p. 71°.

o-*Chloropropionanilide*, $C_6H_4Cl{\cdot}NH{\cdot}COC_2H_5$, colourless plates, m. p. 91°.

Chloroimino-o-chloropropionanilide, $C_6H_4Cl{\cdot}NCl{\cdot}COC_2H_5$, colourless plates, m. p. 57°.

Bromoimino-o-chloropropionanilide, $C_6H_4Cl{\cdot}NBr{\cdot}COC_2H_5$ pale yellow prisms, m. p. 106°.

2:4-*Dichloropropionanilide*, $C_6H_3Cl_2{\cdot}NH{\cdot}COC_2H_5$, colourless prisms, m. p. 121°.

Chloroimino-2:4-dichloropropionanilide, $C_6H_3Cl_2{\cdot}NCl{\cdot}COC_2H_5$, colourless plates, m. p. 64°.

Bromoimino-2:4-dichloropropionanilide, $C_6H_3Cl_2{\cdot}NBr{\cdot}COC_2H_5$, pale yellow rhombs, m. p. 66°.

2:4-*Trichloropropionanilide*, $C_6H_2Cl_3{\cdot}NH{\cdot}COC_2H_5$, colourless prisms, m. p. 161°.

Chloroimino-2:4:6-trichloropropionanilide, $C_6H_2Cl_3{\cdot}NCl{\cdot}COC_2H_5$, pearly plates, m. p. 80°.

Bromoimino-2:4:6-trichloropropionanilide, $C_6H_2Cl_3{\cdot}NBr{\cdot}COC_2H_5$, bright yellow plates, m.p. 106°.

*44. "*The Constitution of the Metallic Cyanides as deduced from their Synthetic Interactions: the Constitution of Hydrogen Cyanide.*" By J. WADE.

The formation of both nitriles and isocyanides from metallic cyanides has been explained in three ways: (1) the metallic cyanides are iso-cyanides, and yield iso-cyanides by direct interchange; nitriles are formed subsequently by isomeric change; (2) hydrogen cyanide is tautomeric and forms two classes of metallic salts (Laar); (3) the metallic cyanides are isocyanides and yield iso-cyanides as in (1); nitriles are formed when the metal is highly positive by the intervention of additive compounds, such as $K{\cdot}N{:}Cl{\cdot}Et = KI + N{:}C{\cdot}Et$ (Nef).

Further investigation of the conditions under which isocyanides are transformed into nitriles shows that the first of the above hypotheses is inadequate. The synthetic cyanides are thus independent and primary products. The tautomerism of hydrogen cyanide does not, however, account for the facts observed; these would necessitate the tautomerism of the metallic cyanides.

The third or additive hypothesis accounts for the dual action of potassium and similar cyanides. There is no evidence, however, that the action between silver cyanide and alkyl haloids is ionic in character, nor does the hypothesis account for the different action of alkyl and acyl haloids on silver and similar cyanides.

The action of alkyl iodides on silver cyanide is parallel to their action on alkyl isocyanides, the alkyl in each case

combining with the nitrogen of the cyanogen group, whereas in the action of acyl halogen compounds the acyl combines with the carbon. The alkyl radicles are feebly electropositive, the acyl electronegative; the additive power of the unsaturated carbon of the isocyanides is thus dependent not merely on the electrochemical character of the attached radicle, as Nef postulates, but on its electrochemical position relatively to the radicle of the interacting halogen compound.

When the metal or alkyl of the isocyanide is sufficiently positive with regard to the radicle of the interacting molecule, the latter combines with the carbon of the isocyanogen group, but when the two radicles are electrochemically adjacent, the interacting molecule combines with the nitrogen:—

$$\left\{\begin{array}{l} K\cdot N{:}C + EtI = K\cdot N{:}CI\cdot Et = KI + N{:}C\cdot Et \\ K\cdot N{:}C + EtKSO_4 = K\cdot N{:}C(SO_4K)\cdot Et = K_2SO_4 + N{:}C\cdot Et. \\ \quad + \qquad\quad + \\ \text{strongly} \quad \text{feebly} \end{array}\right.$$

$$\left\{\begin{array}{l} Et\cdot N{:}C + EtI = Et\cdot NEtI{:}C\ (\rightarrow NHEt_2) \\ Ag\cdot N{:}C + EtI = Ag\cdot NEtI{:}C = AgI + NEt{:}C \\ \quad + \qquad\quad + \\ \text{feebly} \quad \text{feebly} \end{array}\right.$$

$$\left\{\begin{array}{l} Et\cdot N{:}C + AcCl = Et\cdot N{:}CCl\cdot Ac\ (\rightarrow CH_3\cdot CO\cdot CO_2H) \\ Ag\cdot N{:}C + AcCl = Ag\cdot N{:}CCl\cdot Ac = \\ \qquad\qquad = AgCl + N{:}C\cdot Ac\ (\rightarrow CH_3\cdot CO\cdot CO_2H) \\ Cu\cdot N{:}C + PhN{:}N\cdot Cl = Cu\cdot N{:}CCl\cdot N{:}NPh = \\ \qquad\qquad = CuCl + N{:}C\cdot N{:}NPh. \\ \quad + \qquad\quad - \\ \text{feebly} \quad \text{strongly} \end{array}\right.$$

The principle of this hypothesis is applicable to other similar cases of dual action.

The reactions which indicate an isocyanic constitution for hydrogen cyanide are also susceptible of an opposite interpretation. The physical and physiological properties of hydrogen cyanide also correspond with those of the nitriles.

It is probable that the metallic cyanides are, as Gautier suggested, salts of a labile hydrogen isocyanide; their relation to ordinary hydrogen cyanide or formonitrile is similar to that of the salts of Hantzsch's labile *iso*nitromethane to normal nitromethane—

$$CH_3\cdot NO{:}O + K\cdot OH = CH_3\cdot NO(OH)\cdot OK = \\ = H_2O + CH_2{:}NO\cdot OK;$$

$$H\cdot C{:}N + K\cdot OH = H\cdot C(OH){:}NK = H_2O + C{:}NK.$$

The labile isomeride could only be made from metallic cyanides by the action of acids. These necessarily contain electronegative radicles, and therefore combine with the carbon of the cyanide, yielding the normal or nitrilic hydrogen compound—

$$K\cdot N{:}C + HCl = K\cdot N{:}CH\cdot Cl = KCl + N{:}CH.$$

*45. "*The Absorption Spectra of Metallic Nitrates.*" Part I. By W. N. Hartley, D.Sc., F.R.S.

Soret was the first to observe an absorption band in solutions of nitric acid and potassium nitrate, but Soret and Rilliet did not observe it in ethyl nitrate. The author made, in 1887, an extended examination of the absorption spectra of nitrates of the simplest constitution to see if he could find any difference corresponding to that observed by Bunsen in the salts of didymium (*Pogg. Ann.*, 1866, cxxvii., 103).

The results obtained by the author show that the curves of nitric acid and potassium nitrate are identical, but those from silver and thallium nitrates differ widely from one another and from that of nitric acid. In the case of nitric acid, the mean position of the band primarily due to NO_3 lay between λ 317 and 285. The general effect of silver nitrate is to cause an absorption of all rays beyond λ 340, but there is a feeble transmission of a line about λ 274. Thallium nitrate has an absorption band from λ 324 to 274. With a dilution of *N*/1000, there is still an absorption of the rays about λ 242, although water transmits the rays to λ 211.

The solutions were examined in two ways, namely—(1) by using a fixed length of tube (200 m.m.) and varying the strength of the solutions, the best results being obtained with solutions between *N*/20 and *N*/200; (2) by using normal solutions and varying the length of tube.

With normal solutions and a 200 m.m. tube nitrates of the following transmitted a strong continuous spectrum to somewhere about λ 340, after which there was a complete general absorption.

H λ346	Mg λ357	Tl λ340 (*N*/4)
Li 346	Ca 340	Ba 340 (*N*/4)
Na 340	Zn 346	Pb 346 (*N*/2)
K 346		
Ag 355		

46. "*A Method of Determining the Ratio of Distribution of a Base between Two Acids.*" By H. M. Dawson and F. E. Grant.

The method consists in shaking up an aqueous solution containing two acids and a base (the latter being insufficient for saturation of the acids) with a second liquid which is but slightly miscible with water, and which is capable of extracting one and one only of the four substances present in the solution. Tartaric, malic, succinic, and citric acids are not extracted from their aqueous solutions by chloroform, but considerable quantities of acetic acid are.

Determinations of the ratio of distribution of acetic acid between water and chloroform at 20° have been carried out, and show that this ratio increases very considerably with the dilution. By means of a diagram on which the acetic acid concentrations are plotted as abscissæ, and the distribution coefficients as ordinates, the concentration of the acids in water corresponding to any given concentration in chloroform can be readily ascertained. The influence of the presence of the salts and the second acid on the distribution ratio of the acetic acid has been investigated and found to be small.

On shaking up an aqueous solution containing soda, acetic and tartaric acids, with chloroform, and determining the concentration of the acetic acid in the latter, then from the diagram the concentration of the free acetic acid in the aqueous solution can be ascertained. If the original concentrations of the base and the two acids are known, the concentrations of the free tartaric acid and of the two salts can be calculated.

The aqueous solutions were prepared in such a manner that after shaking up with an equal volume of chloroform, the base and the two acids were present in as nearly as possible equivalent quantities, the excess of acetic acid required in the original solution being determined from the preliminary distribution experiments.

Several series of experiments have been carried out in which acetic acid is compared with the several acids above mentioned, three or four concentrations being investigated in each case, and the ratios determined for the distribution of the base between acetic and each of these acids.

This method of determining the ratio of distribution of a base between two acids can also be employed where the second liquid takes up two of the components, for example, the two acids from the aqueous solution, but the ratios of distribution of the two acids between water and the second liquid must differ considerably from one another at any given concentration.

47. "*On the Molecular Complexity of Acetic Acid in Chloroform Solution.*" By H. M. Dawson.

The considerable increase of the ratio in which acetic acid distributes itself between water and chloroform with increasing dilution is shown to be due to a gradual dissociation of the double molecules present in the latter solvent into simple molecules.

The limiting concentrations at which distribution experiments have been carried out correspond to an almost complete change in the molecular complexity of the dissolved acid. At a concentration of 12 grms. per litre of chloroform, the acetic acid present in the form of double molecules is about five times as great as that in the form

of simple molecules, whilst at a concentration of 0·1 grm. per litre the proportion is only one-fifth.

The formation of double molecules by substances containing the hydroxyl group, when dissolved in liquids not containing this group, appears to be essentially dependent upon the concentration.

48. "*On the Existence of Polyiodides in Nitrobenzene Solution.*" By H. M. Dawson and R. Gawler.

In the course of experiments on the ratio of distribution of iodine between potassium iodide solution and nitrobenzene, it was observed that as the amount of iodine added was continually increased, the concentration of the iodine in the aqueous layer reached a maximum value and then gradually decreased.

Further distribution experiments, made with a view of explaining this peculiar phenomenon, showed that the potassium iodide in the aqueous solution was extracted by the nitrobenzene on addition of iodine to the system. Using the same volumes of nitrobenzene and potassium iodide solution in successive experiments, the amount of potassium iodide thus extracted by addition of varying quantities of iodine was determined, and further, the influence of the amount of nitrobenzene for constant quantities of potassium iodide solution and of iodine was investigated. The removal of the potassium iodide by the nitrobenzene is probably due to the formation of polyiodides and the great solubility of the latter in nitrobenzene.

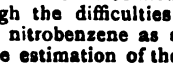
With a view of determining the nature of the polyiodides thus formed, the solubility of potassium iodide in nitrobenzene containing different quantities of iodine has been determined. If the concentration of the iodine does not exceed 0·3 grm.-molecules per litre, it is found that one molecule of potassium iodide dissolves for each molecule of iodine present, indicating the formation of the triiodide. As the concentration of the solutions thus saturated with regard to potassium iodide increases, the molecular ratio of iodine to potassium iodide gradually increases and approximates to the value two.

Determinations have also been made of the solubility of iodine in nitrobenzene containing potassium iodide. For all solutions containing more than sixty grms. of potassium iodide per litre, the molecular ratio of the dissolved iodine to potassium iodide is equal to four, and there is some probability that this ratio holds for the more dilute solutions if the uncombined iodine present in the solution is taken into account. The results of this series of solubility determinations lead to the conclusion that the polyiodide KI_9 is present in the solution.

The nitrobenzene solutions have been examined with regard to their electrical conductivity, and in accordance with the high dielectric constant of nitrobenzene it is found that the solutions are good conductors. Their conductivity is approximately one-fifth that of aqueous solutions of potassium iodide of the same molecular concentration. Determinations of the freezing point of solutions of iodine in nitrobenzene, and of solutions containing in addition to iodine an equimolecular amount of potassium iodide, indicate that the triiodide is dissociated to a very considerable extent, although the difficulties of freezing point measurements with nitrobenzene as solvent prevent an accurate quantitative estimation of the degree of dissociation.

49. "*Derivatives of α-Aminocamphoroxime.*" By A. Lapworth and A. W. Harvey.

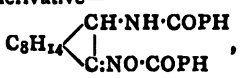
α-Aminocamphoroxime—



is made by heating aminocamphor with a solution of hydroxylamine acetate in presence of a large excess of sodium acetate. It crystallises from benzene in flattened prisms or thin plates melting at 144—145° and dissolves in dilute alkalis and acids. It has $[\alpha]_D = +60{\cdot}5°$ in a 1 per cent solution in absolute alcohol and $+36{\cdot}7°$ in dilute acids.

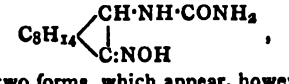
The *hydrochloride*, $C_{10}H_{18}N_2O_3,HCl,H_2O$, forms small prisms and has $[\alpha]_D = +38{\cdot}3°$ in water. The *platinichloride*, $(C_{10}H_{19}N_2O)_2PtCl_6$, forms yellow needles melting and decomposing at 209—211°.

The *dibenzoyl* derivative—



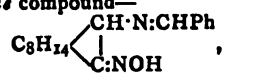
crystallises in brilliant prisms which melt at 146—147°. In absolute alcohol $[\alpha]_D = +104{\cdot}8$.

The *carbamide*—



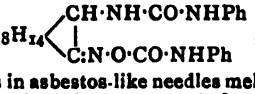
crystallises in two forms, which appear, however, to melt and decompose at the same temperature, namely, 203—204°. It has $[\alpha]_D = +40{\cdot}9°$ in absolute alcohol.

The *benzylidene* compound—



prepared by the interaction of the oxime with benzaldehyde in alcoholic solution, forms brilliant, transparent, apparently hemimorphic prisms melting at 153—154°. In alcohol $[\alpha]_D = 226{\cdot}9°$.

Aminocamphoroxime unites with two molecular proportions of phenylcarbimide, forming a *compound*—



which crystallises in asbestos-like needles melting at 175—177°. In absolute alcohol $[\alpha]_D = -56{\cdot}6°$.

The products obtained by the action of heat or of dehydrating agents, such as acetyl chloride, upon amino camphoroxime do not appear to contain any compound corresponding with the campholenonitriles.

50. "*Preparation of Sulphamide from Ammonium Amidosulphite.*" By E. Divers and M. Ogawa.

It has been pointed out by the authors (*Trans.*, 1900, lxxvii., 324) that a substance, apparently sulphamide, is one of the products of decomposition of ammonium amidosulphite. They have since proved that this substance is sulphamide, and that ammonium amidosulphite is a much better source of it than sulphuryl chloride has been found to be by Hantzsch and Holl (*Ber.*, 1901, xxxiv., 30). The yield is 10 per cent or more, and it is accompanied by no substances which are difficult to remove. Ammonium amidosulphite is slowly heated to, and then maintained at, a temperature of about 70°, so as first to decompose the salt and then to destroy much of the thionic compounds which accompany the sulphamide. The product is dissolved in water and practically everything but ammonium amidosulphate separated from the sulphamide by treating the solution with barium hydroxide and with silver nitrate. With some simple precautions, necessitated by the presence of the amidosulphate, the sulphamide is precipitated as silver salt and recovered from this, almost as Traube directs.

51. "*Hypoiodous Acid.*" By R. L. Taylor.

It has been concluded by Orton and Blackman (*Trans.*, 1900, lxxv., 830) that "the solutions obtained from iodine and mercuric oxide contain only a small quantity of hypoiodite, and that the iodine is chiefly present as iodate." This is contrary to the results described by the author in a former paper (*Mem. Manchester Phil. Soc.*, 1897, xli., 8), where he used aqueous iodine (1 part in 5000), and obtained 80 to 90 per cent of the possible amount of hypoiodite. He has made further experiments and finds that the proportion of iodine existing in the filtrate as hypoiodous acid increases with the fineness of division of the iodine, and diminishes as the proportion of iodine to the water employed increases, and with the time occupied in shaking and filtering. Using powdered

iodine, about 2 parts to 1000 of water, and occupying (as Orton and Blackman did) fifteen minutes in shaking and ten minutes in filtering, the author obtains results not very different from theirs; but using *precipitated* iodine with the same proportion of water, and taking only a little over a minute in shaking and filtering, the filtered liquid contains 44 to 52 per cent of the possible amount of iodine as hypoiodous acid, and of the total iodine in the filtered liquid 90 to 95 per cent exists as hypoiodous acid, and only 5 to 10 per cent as iodic acid. The solution of hypoiodous acid decomposes very rapidly, beginning to turn brown, owing to the liberation of iodine, almost immediately after filtering, unless a very dilute solution of iodine has been employed. Consequently, it is impossible to obtain good results unless all the operations are performed very quickly. The filtered liquid always contains a little mercury. The author has estimated this, and finds that although the hypoiodite in the filtrate may exist partly as mercuric hypoiodite, there is so little mercury present that a considerable proportion of the iodine must exist as hypoiodous acid.

52. "*Synthesis of Imino-ethers. N-aryl Benziminoethers.*" By G. D. Lander.

The preparation of imino-ethers of the type Ph·COR:NPh from the imide chlorides of the corresponding benzoyl arylamines was described. The reaction between the imide chlorides and sodium alcoholates occurs most readily when the latter are employed in alcoholic solution. Intra-molecular re-arrangement does not take place in the formation of the methyl compounds.

N-phenylbenziminoethyl ether, $C_6H_5C(OEt):NC_6H_5$, boils at 168—170° at 14 m.m.; the *methyl* compound at 157—158° at 12 m.m., and the n-*propyl* ether at 177—179° at 11 m.m.

As a by-product in the synthesis of the methyl and ethyl ethers, benzoyldiphenylbenzenyl amidine,—

$$C_6H_5C:NC_6H_5\cdot N(COC_6H_5)C_6H_5,$$

yellow prisms of m. p. 170—172°, was detected.

N-o-tolylbenziminoethyl ether, $C_6H_5C(OEt):NC_7H_7$, boils at 179—180° at 15 m.m., the *methyl* ether at 173° at 15 m.m.

N-p-tolylbenziminoethyl ether boils at 178° at 11 m.m., the *methyl* ether at 177° at 12 m.m.

Reference was made to the formation by the action of acetyl chloride containing hydrochloric acid on a light petroleum solution of the base, of the hydrochloride of *N-o*-tolylacetiminoethyl ether, $CH_3C(OEt):NC_7H_7,HCl$, which is crystalline, melts at 109—110°, and decomposes into ethyl chloride and acetotoluidide. Similarly, *N-phenylacetiminoethyl ether hydrochloride*,—

$$CH_3C(OEt):NC_6H_5,HCl,$$

m. p. 100° with decomposition, has been prepared for the first time.

*53. "*Nitration of s-Trihalogenacetanilides.*" By K. J. P. Orton.

As a sequel to the author's work on the action of nitric acid on *s*-trihalogenanilines (*Proc.*, xviii., 58) the nitration of *s*-tribromoacetanilide and of 4-chloro-2 : 6-dibromoacetanilide has been investigated. Remmers (*Ber.*, 1874, vii., 351) and Bentley (*Amer. Chem. Journ.*, 1898, xx., 472) have subjected *s*-tribromoacetanilide to the action of fuming nitric acid; the former obtained an anilide, $C_6HBr_3(NO_2)NHAc$, crystallising in yellow needles from which an aniline, $C_6HBr_3(NO_2)\cdot NH_2$, was prepared by hydrolysis with ammonia; it crystallised in yellow needles, and melted at 214—215°, whilst 2 : 4 : 6-tribromo-3-nitroaniline, according to Körner (*Jahresberichte*, 1875, 347), melts at 102·5°. Bentley was unable to confirm Remmer's results.

By cautiously warming the anilides above mentioned with fuming nitric acid, the author has obtained respectively 2 : 4 : 6-tribromo-3-nitroacetanilide and 4-chloro-2 : 6-dibromo-3-nitroacetanilide. In neither case was the nitration complete; the nitrated anilide was isolated in a pure state by conversion of the mixture of anilides into the acetyl-chloroamino-derivatives; the latter can be readily separated by fractional crystallisation from petroleum, in which the nitrated chloroamine (for example, acetyl-chloroamino-2 : 4 : 6-tribromo-3-nitrobenzene) is but little soluble. By treatment with alcohol, the anilide is obtained from the pure acetylchloroamino-derivative.

The following substances have been prepared:—

2 : 4 : 6-*Tribromo-3-nitroacetanilide*, $C_6HBr_3(NO_2)NHAc$, from 2 : 4 : 6 tribromo-3-nitroaniline (m. p. 102°, Körner), and by nitrating *s*-tribromoacetanilide, colourless needles, m. p. 216—217°; 2 : 4 : 6 - *tribromo-3-nitrodiacetanilide*, $C_6HBr_3(NO_2)\cdot NAc_2$, rhombs, m. p. 175—176°; *acetylchloroamino* 2 : 4 : 6 *tribromo-3-nitrobenzene*,—

$$C_6HBr_3(NO_2)NClAc,$$

prisms, m. p. 159°.

4-*Chloro*-2 : 6-*dibromo-3-nitroaniline*,—

$$C_6HClBr_2(NO_2)\cdot NH_2,$$

prepared by brominating 4-chloro-3-nitroaniline (m. p. 103°), and by hydrolysis of the corresponding anilide, pale yellow needles, m. p. 103—104°.

4-*Chloro*-2 : 6-*dibromo-3-nitroacetanilide*,—

$$C_6H\cdot ClBr_2(NO_2)\cdot NHAc,$$

prepared from the corresponding aniline and by nitrating 4 chloro-2 : 6-dibromoacetanilide, silky needles, m. p. 224°; *acetylchloroamino-4-chloro-2 : 6-dibromo-3-nitrobenzene*, $C_6HClBr_2(NO_2)\cdot NClAc$, short prisms, m. p. 134—135°.

Attention is called to the fact that, whereas in the action of nitric acid on *s*-tribromoaniline a nitroamine is first formed, which then undergoes change, the nitro-group displacing the para-bromine atom in the nucleus (*Proc.*, xviii., p. 58), with the anilide no such displacement occurs, but only nitration in the meta-position. Acetylation, combined with the presence of two ortho-placed halogen atoms, prevents partially, at least, the amino group from exercising its usual directing function.

54. "*Purpurogallin.*" Preliminary Notice. By A. G. Perkin and A. B. Steven.

To purpurogallin, an oxidation product of pyrogallol, Clermont and Chautard (*Jahresbericht*, 1882, 683) assigned the formula $C_{20}H_{16}O_9$, and described a tetra-acetyl compound, $C_{20}H_{12}O_9(C_2H_3O)_4$, m. p. 186°, and a tetrabromo-derivative, $C_{20}H_{12}Br_4O_9$, m. p. 202—204°. Nietzki and Steinmann (*Ber.*, 1887, xx., 1278) preferred the formula $C_{18}H_{14}O_9$, and found that on distillation with zinc dust naphthalene is produced. This preliminary investigation, while corroborating, as a rule, the analytical numbers of these chemists, indicates that the molecular weight of purpurogallin is much lower, most probably being represented by $C_{11}H_8O_5$. The *tetra-acetyl* compound, m. p. 182—183°, $C_{11}H_4O_5(C_2H_3O)_4$, and the *tribenzoyl* compound, $C_{11}H_5O_5(C_7H_5O)_3$, minute colourless prisms, m. p. 213° (found, C = 71·99; H = 3·86; molecular weight [cryoscopic] = 556), have been studied. Analyses of the *dibromo*-compound, $C_{11}H_6Br_2O_5$, m. p. 204—206° (found, C = 35·10; H = 1·67), and the *monopotassium* salt, $C_{11}H_7O_5\cdot K$ (found, K = 14·76), minute brown-violet needles, point to the correctness of this formula. Hooker (*Ber.*, 1887, xx., 3260), by means of nitrous acid, obtained purpurogallin from gallic acid. If, however, potassium ferricyanide in conjunction with potassium acetate be employed, a small quantity of a new compound crystallising in minute orange-red needles is formed, which appears to be purpurogallin carboxylic acid, $C_{11}H_7O_5\cdot CO_2H$ found, C = 54·56, 54·14; H = 3·73, 3·18).

55. "*Quercetagetin.*" By A. G. Perkin.

Quercetagetin was isolated from the flowers of the African marigold (*Tagetes patula*) by Latour and Magnier de la Source (*Bull. Soc. Chim.*, 1877, xxviii., 337) as a yellow crystalline substance, $C_{27}H_{22}O_{13}+4H_2O$. A re-investigation indicates that its true formula is $C_{15}H_{10}O_8$ (found, C = 56·66, 56·47; H = 3·25, 3·31), as it gives a *sal-*

phate, $C_{15}H_{10}O_8,H_2SO_4$ (found, C = 43·28; H = 3·29), orange needles, and a *monopotassium* salt, $C_{15}H_9O_8K$ (found, K = 10·74). Quercetagetin melts at 318—320°, gives an *acetyl* compound, $C_{15}H_4O_8(C_2H_3O)_6$ (found, C = 57·37; H = 3·87; $C_{15}H_{10}O_8$ = 55·82), colourless needles, m. p. 203—205°, and dyes shades browner, though somewhat similar to quercetin, to which class of colouring-matter it appears to belong. It contains no methoxyl group, and on fusion with alkali gives protocatechuic acid and a phenol at present unidentified. As there is considerable difficulty in obtaining raw material, the conclusion of the investigation may be delayed for some time.

NOTICES OF BOOKS.

The Elements of Physical Chemistry. By HARRY C. JONES, Associate Professor of Physical Chemistry in the Johns Hopkins University. London: Macmillan and Co., Ltd. New York: The Macmillan Company. 1902. Pp. 565.

WITHIN the last fifteen years a new branch of science has come into existence, occupying a position between physics and chemistry, and known as physical chemistry. The term is not new, though the new science is different to the old.

In the author's opinion it is not necessary to reject or ignore the work of such men as Gladstone, Bunsen, Kopp, and Regnault in order to appreciate the gigantic strides made by the *new* physical chemistry; it is in their work that lies the foundation of much that has been developed within the last few years.

No one can use this book successfully without an elementary knowledge of physics, chemistry, and mathematics, and it would be well to add the elements of thermodynamics and differential equations.

The new physical chemistry really begins with the chapter on solutions; the discovery of the relations between dilute solutions and gases has placed the subject of solutions at the very foundation of the new developments in physical chemistry.

Matter is known in three different states—solid, liquid, and gaseous; and as matter in every state can be mixed with matter in every other state, it is obvious that we can have nine different classes of solutions. These are—gas in gas, liquid in gas, solid in gas, gas in liquid, liquid in liquid, solid in liquid, gas in solid, liquid in solid, solid in solid; and it is stated in advance that well-defined examples of all these classes are known. In this chapter, which fills over 100 pages, the author considers solutions in all their bearings; osmotic-pressure, the relations between osmotic-pressure and gas-pressure, the theory of electrolytic dissociation, the lowering of the freezing-point of solvents by dissolved substances, diffusion, colour, and other properties of solutions.

Little or nothing was heard of solid solutions until van't Hoff published his now well-known paper about eleven years ago. When some substances are dissolved in solvents other than water they give abnormally small depressions of the freezing-point. After studying and experimenting on this subject van't Hoff was led to suspect that when certain solutions are frozen the solid which separates is not the pure solvent, but a mixture of the solvent and the dissolved substance, forming a solid solution. If a solid solution is a solid homogeneous complex of several substances, in which the properties can change without destroying the homogeneity, then examples are known, such as the alums and the formation of "mixed crystals" which are to be distinguished from double salts.

A striking example of diffusion in solids where no chemical action takes place is the penetration of hot porcelain by carbon; when a porcelain crucible is heated in graphite the carbon completely penetrates the porcelain; zinc objects covered with a thin layer of copper gradually become white; while, as Sir Wm. Roberts-Austen has lately shown, discs of gold and lead clamped together and kept at a constant temperature of 18° C. for four years, were found to have adhered to each other, whilst the gold had penetrated the lead to a distance of 8 millimetres.

The remainder of the book has largely to do with energy changes, as, in the author's opinion, the fundamental problems of chemistry will never be solved by a study of the material changes alone, but only through elaborate investigations of the energy relations which obtain in systems before reactions, and in the products of the reactions.

The transformations of chemical energy into heat constitute the subject-matter of Chapter VI. on thermochemistry; electrochemistry, Chapter VII., deals with the transformation of chemical energy into electrical, and *vice versâ*. The relations between light and chemical energy furnish the material for Chapter VIII. on photochemistry, and the velocities with which chemical reactions take place, and the conditions of equilibrium in such reactions are examined in Chapter IX., on chemical dynamics and chemical equilibrium; while in Chapter X. measurements of the relative chemical activities of different substances are considered.

It is not too much to say that this is a valuable and important work, and gives in one volume the co-ordinated results of many separate workers. The subject of physical chemistry has already extended into nearly every field of chemical science; it has thrown light on many problems in chemistry, as well as in physics; it has also reached out into biology; and that physical chemistry is to find its way into the geological sciences has become obvious from the work of van't Hoff in the last two or three years.

An Introduction to Modern Scientific Chemistry, in the Form of Popular Lectures Suited for University Extension Students and General Readers. By Dr. LASSAR-COHN, Professor in the University of Königsberg. Translated from the Second German Edition by M. M. PATTISON MUIR, M.A. With 58 Illustrations by the Author. London: H. Grevel and Co. 1901. Pp. 348.

IN this volume the author has endeavoured to give a succinct and accurate demonstration of chemistry on strictly scientific lines, and at the same time in as popular a form as is compatible with the matter and the vast range of the subject.

The form followed in lectures is used as far as possible, and experience has shown that this method adds to the interest of the subject. The book, however, is not divided into numbered chapters, but simply goes on from one subject to another with hardly a break.

The author is lucid in style, and does not overburden his work with an excess of theoretical considerations.

We must, however, remark on the eccentric nature of the illustrations, which, we read, are by the author himself. The lack of perspective in most of them is very noticeable, while throughout they are of the crudest description and are often quite unnecessary. A glance at pages 7, 15, 26, 143, or 176, to mention only a few, will enable the reader to follow our meaning. Otherwise the book is well and clearly printed, and the index is all that can be desired.

Royal Institution.—On Tuesday next, April 8th, Dr. Allan Macfadyen, of the Jenner Institute of Preventive Medicine, will deliver the first of a course of three lectures at the Royal Institution on "Biological Inquiry"; on Thursday, April 10th, Professor Dewar will begin a course of three lectures on "The Oxygen Group of Elements"; and on Saturday Dr. W. H. Cummings, Principal of the Guildhall School of Music, will commence a course of three lectures on "British National Song," with Musical Illustrations. The Friday Evening Discourse on April 11th will be delivered by Professor Dewar on "Problems of the Atmosphere"; and on April 18th by Sir John H. A. Macdonald, his subject being "The Autocar."

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxiv., No. 10, March 10, 1902.

Preparation and Properties of a New Silicon Hydride.—H. Moissan and S. Smiles.—During the solidification of the condensation products resulting from the action of dilute hydrochloric acid on magnesium silicide by means of liquid air, the authors obtained a mixture which after fractionation yields a gas and a liquid body, the latter spontaneously inflammable in air. The gas they intend to further investigate. The liquid compound contains for one molecule of silicon a molecule and a-half of hydrogen. To this they attribute the formula Si_2H_6, by analogy with ethane gas, and they hope to be able to verify this theory and to establish the vapour density of this new compound.

Researches on the Transformations of Steel by the Dilatometric Method.—Georges Charpy and Louis Grenet.—Using the dilatometric method, two transformations are observed in iron-carbon alloys:—(1) A sudden change taking place at 700°, with contraction of volume; this corresponds to the absorption of heat observed at the critical-point a_1 in the pyrometric method. (2) A gradual change, apparently corresponding to a contraction for steels containing more than 0·85 per cent of carbon, and to a dilatation for steels containing more than 0·85 of carbon and ending at a temperature approaching that of the critical-point a_3, as indicated by the pyrometric method. The critical-point a_2, which is observed towards 750° by the pyrometric method, apparently corresponds to no variation in the phenomenon of dilatation.

Action of Hydrogen Peroxide on Zinc Oxide.—M. de Forcrand.—The author's researches on the peroxidation of zinc oxide by means of peroxide of hydrogen lead him to the following results:—(1) There exists no oxide of zinc intermediate between ZnO and Zn_3O_5, and consequently the products obtained by Thénard and Kouriloff are mixtures. The same is also true of Haass's oxide, Zn_5O_8. (2) There exist three different degrees of peroxidation of zinc, Zn_3O_5, Zn_4O_7, and ZnO_2; the first is stable at 100°, the last is extremely unstable, even in the cold. (3) These bodies are all hydrated, and permanently retain at least as many molecules of water as atoms of oxygen in excess, in such a manner that they may just as well be considered as compounds of hydrogen peroxide and the protoxide (anhydrous or hydrated) as hydrated peroxides.

A New Sodium Phosphate.—H. Joulie.—The author shows that there exists a phosphate of sodium intermediate between monosodic and disodic phosphates which might be called the sesqui-sodic phosphate, and which corresponds to the formula $3NaO,3HO.2PO_5$. This phosphate differs from disodic phosphate (1) in being much more soluble in water and much more easily preserved in the form of a concentrated solution without crystallising; (2) in medicine it is active in much smaller doses; (3) it has a slight and not unpleasant taste; (4) it will probably be able to be employed for hypodermic injections in more concentrated solutions than disodic phosphate.

Reduction of the Ortho-nitrated Azoic Colouring-matters. Production of Substituted Derivatives of Phenyl-pseudo-azimidobenzol.—A. Rosenstiehl and E. Suais.—When azoic colouring-matters derived from para- and meta-nitramines are reduced, it is possible to obtain the nitramines, azoxyamines, and the corresponding free azoamines or the azoic colouring-matters derived from the azoxyamines or phenylenediamines, according to the nature of the reducing agent employed. The reactions are often quantitative. With azoic colouring-matters derived from orthonitramines a different reaction takes place. According as the reducing agent is an alkaline solution of glucose or of a sulphide, a single product is obtained less sensitive to an excess of the reducing agent. The reaction is general; the authors establish it for orthonitraniline and the isomeric orthonitrotoluidines.

Variation in Rotatory Power of Stable Left-hand Ether Salts of Borneol.—J. Minguin and E. Grégoire de Bollemont.—The authors examine the influence of the substitution of certain radicles for the hydrogen atom of OH. For this purpose they prepare from the stable left-hand isomer (borneol $[\alpha]_D = -38{\cdot}09°$) a series of ether salts. Their properties with regard to rotatory power, boiling-point, &c., are given in a table.

Bulletin de la Société Chimique de Paris. Series 3, Vol. xxv., No. 15.

Active Erythrites.—L. Maquenne and G. Bertrand.—Already noticed.

Racemic Erythrite.—L. Maquenne and G. Bertrand.—Already noticed.

Some New Derivatives of Dialcoylamidobenzoylbenzoic and Tetrachlorised Dialcoylamido-*m*-oxybenzoylbenzoic Acids. The Dialcoylamidised Anthraquinones and the corresponding Dialcoylamidised Oxyanthraquinone.—MM. Haller and Umbgrove.—The authors have already given the preparation of the tetrachlorised dimethyl- and diethylamidobenzoylbenzoic acids, as well as that of a certain number of their ethers and their mixed anhydrides. By reacting with nascent nitrous acid, or nitric acid, on the dimethylamidised acid, they have obtained the nitroso and nitrated derivatives.

The Synthesis of Hexamethyltriamidodiphenylenephenylmethane and of the Colouring Material derived from it.—A. Haller and A. Guyot.—Already noticed.

On Hydrochlorate of Urea from Phenylhydrazine.—P. Cazeneuve.—In a paper on the acid molecular compounds of urea from phenylhydrazine (diphenylcarbazide), the author described a series of combinations with the organic acids, but he was unable to obtain the hydrochloric compound of this body any more than the nitric or sulphuric compounds. However, the hydrochloric compound can be obtained under the following conditions:—Five grms. of urea are dissolved in 60 to 80 c.c. of hot alcohol at 93°. Into this alcoholic solution, when nearly cold, a large excess of anhydrous ether saturated with hydrochloric acid gas is poured. Gradually the hydrochlorate of urea crystallises out in small crystals having the appearance of cauliflowers. This body is white and insoluble in water or ether, but soluble in hot alcohol. It deteriorates in boiling water. It is not decomposed *in vacuo* over soda-lime, and melts with decomposition at 125°. The estimation of the hydrochloric acid, carried out with a titrated solution of soda on the substance dried *in vacuo*, gave the following results:—Substance taken, 0·595 grm.; HCl = 0·07738 grm., or 12·97 per cent Cl. The formula $CO<^{NH.NH.C_6H_5}_{NH.NH.C_6H_5}$ HCl requires 12·94 per cent. Ordinary urea also gives a mono-hydrochloric compound.

The Chromised Violet Colouring-matters derived from Diphenylcarbazide.—P. Cazeneuve.—The compound specially dealt with in this paper was prepared in the following manner:—Twelve grms. of urea from phenylhydrazine were dissolved in 25 grms. of hot acetic acid and 25 grms. of water. Chromic acid in equimolecular proportion was gradually added; that is, 5 grms. dissolved in 50 grms. of water. Carbonic acid is given off, and at the same time the liquid takes a deep violet tint. On cooling, most of the colouring material is deposited.

The supernatant liquid is decanted. The residue is taken up with hot alcohol, and the colouring material is again deposited on cooling. This is repeated several times. The author then goes on to describe the various properties and reactions of this body. Another similar colouring-matter was obtained by using a much smaller quantity of chromic acid; the resulting body contains only 0·93 per cent of chromium, against 5·62 or 5·5 in the former one. These two materials, however, possess the same properties with regard to dyeing.

The Use of Diphenylcarbazide for the Detection of Chromic Acid in Cotton Dyed with Chrome Yellow.—P. Cazeneuve.—Diphenylcarbazide is the most delicate reagent for chromic acid, without the possibility of confusion with any other metallic body; its sensitiveness reaches even more than 1 part in 1,000,000. Any chromate in aqueous solution, slightly acidulated with acetic or hydrochloric acid, gives an intense violet colouration with diphenylcarbazide in the form of powder. For testing cotton goods, the material is placed in a test-tube with 1 c.c. of a $\frac{1}{10}$th per cent solution of potash. The threads are instantly bleached in the case of chromate of lead or chrome yellow. The liquid is then made strongly acid with acetic acid. A few grains of diphenylcarbazide or of acetate of diphenylcarbazide added to the liquid develop, on agitation in the cold, a beautiful violet colouration. This reaction is trustworthy, sensitive, and rapid.

Characteristics of Essences of Orange Flowers from the 1901 Crop.—Eug. Theulier.—The author gives, in the form of a table, a number of physical constants, proportions of ethers, and anthranilate of methyl observed in a large number of samples taken from the daily distillations from May 16 to June 2, 1901.

MISCELLANEOUS.

Oxidation by the Oxygen of the Air.—H. Biltz.—The slow oxidations brought about by the oxygen of the air, in the presence of water, are accompanied by a secondary formation of peroxide of hydrogen. Examples of this action, first observed by Schoenbein, are not yet very numerous in organic chemistry; the oxidation of the phenylhydrazone of dibromo-*p*-oxybenzaldehyde is, however, a very good example. Two grms. of the phenylhydrazone are dissolved with 3 grms. of potassic hydrate in 50 grms. of alcohol and 30 c.c. of water. A current of air is passed through the solution; after four hours the dilute solution, slightly acidulated with hydrochloric acid, is treated with a large excess of acetate of soda. The osazone thus precipitated is a crystalline powder, soluble in alcohol, fusible at 206°, and having the formula $C_{26}H_{20}N_4Br_4O_2$. In the mother-liquors from the precipitation, diluted to 1 litre, the peroxide of hydrogen can be detected by all the usual reagents; it can be titrated without difficulty with permanganate of potash or iodide of potassium. It can be seen thus that the oxygen of the osazone and that of the peroxide of hydrogen are equal in weight. If, on the other hand, we estimate the amount of oxygen contained in the air used, we find that it is equal to the sum of the oxygen of the osazone, and that of the peroxide of hydrogen together. The oxidation is therefore expressed by the following equation:—
$2C_{13}H_{11}N_2Br_2O + O_2 = C_{26}H_{20}N_4Br_4O_2 + H_2O_2$.—*Berichte*, vol. xxxiii., p. 2295.

Action of Bromine and Sulphide of Carbon on the Methylenic Soda Compounds.—G. Wenzel.—It is well known that iodine transforms soda malonic ether into acetylenetetracarbonic ether by the separation of iodide of sodium and the junction of the two remaining portions of malonic ether. Bromine, in sulphocarbonic solution, reacts differently on this same body. Bromide of sodium is formed, as well as bromide of sulphur, and a compound which crystallises in yellow needles fusible at 139° and having the formula $C_{16}H_{22}S_3O_8$:—
$2C_7H_{11}O_4Na + 2CS_2 + 2Br_2 = C_{16}H_{22}S_3O_8 + 2BrNa + Br_2S$.
The return from the operation is only from 5 to 20 per cent of theory. This body is soluble in alcohol, insoluble in soda, and hardly attacked by acids in the cold. Soda cyanacetic ether undergoes an analogous transformation under the same influences. We obtain a compound, fusible at 225°, of the formula $C_{12}H_{22}N_2S_3O_4$, insoluble in water and ligroin, but soluble in alcohol. The soda compounds of acetylacetone, and of the acetic and phenylacetic ethers will be examined from the same point of view.—*Berichte*, vol. xxxiii., p. 2041.

Action of Fuming Nitric Acid on the Substituted Acrylic Acids.—A. Wahl.—The author has reacted with fuming nitric acid on the crotonate, tiglate, and isolauronolate of ethyl, and on cinnamate of methyl. He describes the methods adopted and the reactions obtained.—*Bull. Soc. Chim.*, Series 3, xxv., No. 16—17.

The Action of Nitric Acid on Anthracene.—O. Dimroth.—The author has elaborated a method of nitration of anthracene which has given him good results. Fifty grms. of finely powdered anthracene are suspended in 200 c.c. of acetic acid, and 20 c.c. of nitric acid at 63 per cent are then added without allowing the temperature to go above 30—35°. The anthracene is dissolved, and the solution very probably contains nitroacetate of anthracene, which, when heated with dilute soda-lye, is transformed into nitroanthracene. If we add fuming hydrochloric acid and acetic acid to the above nitric and acetic solution, while still keeping the temperature down, we obtain chloride of nitroanthracene $C_{14}H_{10}(NO_2).Cl$, which, when digested with dilute soda-lye, also gives nitroanthracene. This compound can be obtained perfectly pure, in yellow prisms fusible at 149°, by a single crystallisation in acetic acid. The above-mentioned chloride occurs in needles fusible at about 163°; if the nitric solution is heated, first on the water-bath and then to boiling, nitric oxide is given off, and a substance deprived of nitrogen is formed which is probably a *dianthranol*—

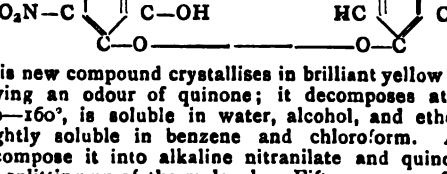
$$HO.C\langle{}^{C_6H_4}_{C_6H_4}\rangle C : C \langle{}^{C_6H_4}_{C_6H_4}\rangle C.OH,$$

or rather a *dianthrone*—

$$OC\langle{}^{C_6H_4}_{C_6H_4}\rangle CH.CH \langle{}^{C_6H_4}_{C_6H_4}\rangle CO.$$

This compound is identical with the product obtained in different manners by Orndorff and Bliss, starting with anthranol.—*Berichte*, vol. xxxiv., p. 219.

Action of Nitrous Acid on Quinone.—J. Schmidt.—By passing nitrous acid fumes produced by the attack of As_2O_3 with HNO_3 through an etherial solution of quinone (10 grs. per 250 c.c.) and cooling to about 78°, a crystalline deposit of *nitranilic-quinone* is obtained—



This new compound crystallises in brilliant yellow prisms having an odour of quinone; it decomposes at about 100—160°, is soluble in water, alcohol, and ether, and slightly soluble in benzene and chloroform. Alkalis decompose it into alkaline nitranilate and quinone by the splitting up of the molecule. Fifteen grms. of liquid nitrous acid placed in a sealed tube with 3 grms. of quinone, reacted violently after twelve hours' contact; the explosion blew the wrought-iron tube enveloping the sealed glass tube into fragments, and these penetrated the walls and doors of the laboratory to a depth of several centimetres.—*Berichte*, vol. xxxiii., p. 3246.

MEETINGS FOR THE WEEK.

MONDAY, 7th.—Royal Institution, 5. General Monthly Meeting.
—— Society of Chemical Industry, 8. "Some Recent Improvements in the Photography of Colour" (Illustrated by Lantern Slides), by E. Sanger Shepherd.

TUESDAY, 8th.—Royal Institution, 3. "Recent Methods and Results in Biological Inquiry," by Allen Macfadyen, M.D., B.Sc., Fullerian Professor of Physiology, R.I.
—— Society of Arts, 8. "Street Architecture," by Prof. Beresford Pite, F.R.I.B.A.

WEDNESDAY, 9th.—Society of Arts, 8. "Ceuta and Gibraltar," by Major-Gen. John F. Crease, C.B.

THURSDAY, 10th.—Royal Institution, 3. "The Oxygen Group of Elements," by Prof. Dewar, M.A., LL.D., D.Sc., F.R.S., Fullerian Professor of Chemistry, R.I.

FRIDAY, 11th.—Royal Institution, 9. "Problems of the Atmosphere," by Prof. Dewar, M.A., LL.D., D.Sc., F.R.S., &c.
—— Physical, 5. "An Apparatus for Vapour-pressure Measurements," by Mr. Grant. "The Use of Cathode Rays for Alternating Current Measurements" and "An Experiment on the Current Growth in an Inductive Circuit," by Mr. Morris. "An Electric Heater," by Dr. R. A. Lehfeldt. "Note on the Compound Pendulum," by S. A. F. White.

SATURDAY, 12th.—Royal Institution, 3. "British National Song" (with Musical Illustrations), by William H. Cummings, Mus.D. (Dub.), F.S.A., Hon R.A.M., Principal of the Guildhall School of Music.

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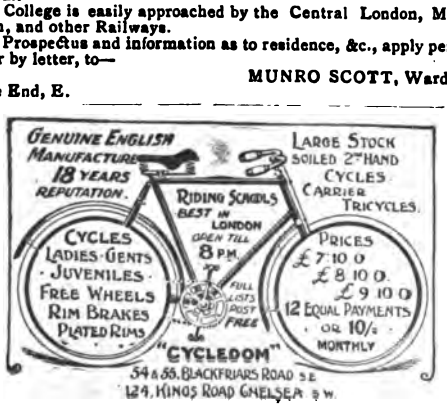
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ON THE RADIO-ACTIVITY OF MATTER.*

By HENRI BECQUERREL, D.C L., Ph.D.,
Member of the Academy of Sciences, Paris.

The property possessed by certain bodies of emitting invisible and penetrating rays was unknown six years ago. The speculations brought about by the experiments of M. Röntgen led to the examination of material bodies, to see if any of them had the power of emitting similar radiations; the phenomenon of phosphorescence naturally was first thought of, being a known method for the transformation and emission of energy. This idea, however, could not be applied to the phenomena with which we are occupied, but it was very fruitful. It led to the choice, among phosphorescent bodies, of the salts of uranium of which the optical constitution is remarkable on account of the harmonic series of the bands of their absorption and phosphorescent spectra. It was while experimenting with these bodies in 1896 that I first observed the new phenomena which I am about to bring before you this evening. I have here the plates of the double sulphate of uranium and potassium, obtained by Lipmann's method, which I used for my first experiments. After having placed one of these plates on the black paper which covered a photographic plate, and leaving it for several hours, I observed on developing the plate that the uranium salt had emitted certain active rays, traversing the black paper, as well as various screens interposed between the plate and the active body, such as thin sheets of glass, aluminium, copper, &c. I soon saw that this phenomenon had nothing to do with phosphorescence, or with any known method of excitation, such as luminous or electric rays, or any appreciable variation of temperature.

I had to deal, therefore, with a spontaneous phenomenon of a new order. The absence of any known exciting cause on a body prepared in the laboratory several years ago, caused me to think that the phenomenon would have been the same at any time it might have been observed; it should therefore be permanent, that is to say, there should not be any appreciable weakening after a very long time. This is, in fact, what I have proved during the past six years. I will show you the first proof I had of the spontaneity of the rays; these rays have traversed the black paper which covered the photographic plate, and a thin strip of copper in the form of a cross. Here again is the radiograph, made about the same time, of an aluminium medal; the unequal absorption of the different thicknesses has caused the appearance of the effigy thereon. After the very first observation I observed that the new radiations would discharge electrified bodies, at some distance in the air, a phenomenon which gives us a second method for studying these rays; the photographic method is specially qualitative, while the electrometer furnishes numerical elements of comparison.

In the course of these first observations, I was led away from the path towards which later experiments brought me back by several facts, of which the following is the principal:—Having protected a photographic plate by means of a sheet of aluminium 2 m.m. in thickness, and having arranged on the aluminium several samples of phosphorescent powders, placed on separate plates of glass, and covered with small tubes like clock shades, the photographic proofs, obtained after forty-eight hours, showed silhouettes of the plates of glass just as if they had been produced by the total refraction and reflection of rays identical with those of light, but which must have traversed the 2 m.m. of aluminium. This photograph is unique; I have never been able to reproduce it or obtain any action with the same sample of sulphide of calcium, nor with any other phosphorescent preparation. At about the same time M. Niewenglowski obtained an impression with sulphide of calcium, and M. Troost with hexagonal blende. To this day I do not know the cause of the activity of these products or its disappearance. These facts, and some others, gave me the idea that the new rays might be a transversal movement of the ether analogous to that of light; but the absence of refraction and a large number of other experiments made me abandon this hypothesis.

In this same year, 1896, I found that all the uranium salts emitted rays of a similar nature, that the radiant property is an atomic one belonging to the element uranium, and electric measurements showed me that metallic uranium was about three and a-half times more active in ionising air than is the double sulphate of uranium and potassium. The same method enables us to study the *rôle* played by the gases in the discharge, and to observe that a sphere of electrified uranium retains its charge *in vacuo*, while in air it loses it. The rate of the fall of potential is sensibly proportional to the potential if the latter is only a few volts; it should be constant and independent of the potential when this is very high. Gas rendered conducting by these rays retains this property for some instants. Between two conductors maintained at constant potentials these rays set up in air a continuous current.

These experiments were taken up and elaborated by Lord Kelvin in 1897, then by Messrs. Beattie and S. de Smolan. In 1899 Mr. Rutherford showed how the phenomena due to the conductivity communicated to gases by uranium, and the existence of a maximum in the current produced, could be explained by the hypothesis of ionisation, to which the beautiful work of Mr. J. J. Thomson has given the seal of authority.

In 1898 M. Schmidt and Mdme. Curie observed, quite independently, that thorium has properties analogous to those of uranium, properties which were specially examined by Mr. Owens and Mr. Rutherford. Mdme. Curie having measured the ionising activity of a large number of minerals containing uranium or thorium, announced the remarkable fact that several minerals were more active than metallic uranium. M. and Mdme. Curie concluded that there must be a more active body than uranium in the mineral, and they undertook the task of isolating it.

By treating one of the most active of these minerals, viz., pitchblende from Joachimstal, they first separated an active bismuth, to which they gave the name of polonium, then shortly afterwards they obtained a very active barium containing a new element, radium.

These bodies are prepared by fractional precipitations, in which one is guided by the indications of the electrometer; the activity of these products is 100,000 times greater than that of uranium.

About the same time M. Giesel succeeded in preparing some very active substances, and in 1900 M. Debierne announced the existence of a new element, actinium, about which, however, we have not heard many particulars. Of all these new bodies radium alone is characterised as a new element; it has an emission spectrum consisting of lines which do not belong to any other known body, and the atomic weight of the active salts of barium was found to increase with the proportion of radium present.

The activity of uranium was not sufficient to excite phosphorescence in other bodies; M. and Mdme. Curie, however, observed this phenomenon with the rays from radium, and further, that the salts of radium were themselves luminous, their luminosity being, like their radio-activity, spontaneous. The activity of radium produces various chemical reactions; it colours glass, it transforms

* A Discourse delivered before the Members of the Royal Institution, Friday, March 7, 1902.

oxygen into ozone, it changes white phosphorus to red, it ionises not only gases but also liquids, such as petroleum and liquid air, and insulating solid bodies, such as paraffin, developing in this latter body a residual conductivity which lasts a long time after the rays have ceased to act. It also causes on organic tissues serious burns analogous to those produced by X rays.

The sample of radium that M. and Mdme. Curie have lent me for the purpose of this lecture enables me to show you a few of these phenomena—ionisation of the air, luminosity, and phosphorescence.

I have observed by means of the photograph I now show, that the radio-activity of polonium will not traverse a thin sheet of black paper forming a small cylinder closed by aluminium or mica, and at the bottom of which was placed the powdered material; the rays from radium easily pass through this envelope; we shall see that still more profound differences exist between these two kinds of rays.

The radio-activity of radium restores to certain crystals, and to glass, the property of becoming phosphorescent by heat, which they have lost owing to a previous elevation of temperature.

The phenomena of absorption, examined either by means of photography, by phosphorescence, or by ionisation of the air, showed the heterogeneity of the class of radiations emitted; subsequent observations have enlarged the field of this research.

Towards the end of the year 1899 M. Giesel, and then MM. Meyer and Schweidler, observed that the rays of radio-active preparations were deviated by a magnetic field in the same manner as are the cathodic rays. For my part, at about the same time, without having heard of these experiments, I observed the same phenomenon with radium. The experiment can be made in the following manner:—A small paper cylinder containing a few grains of the radio-active body is placed horizontally on a photographic plate covered with black paper, between the poles of a magnet; the rays are thrown entirely to one side of the plate. I here show my two first photographs, one of which shows a concentration on one pole of the magnet.

Very shortly afterwards I observed that the rays from polonium are not deviated, and consequently that two kinds of rays exist, one deviable and the other non-deviable. M. and Mdme. Curie have made an electric examination of this subject which has proved the simultaneous existence of these two kinds of rays in the radio-activity of radium, their unequal permeability varying with the distance from the absorbing screens. The accompanying photograph shows these two kinds of rays from radium; I have recently observed that uranium emits only deviable rays, that is, saving the existence of much less active, non-deviable rays. In fact, there does exist a third kind of ray which are not deviable, but are extremely penetrating; they have been shown more particularly by M. Villard.

Thus the activity of radio-active bodies comprises three kinds of rays—rays which are deviable in a magnetic field, which appear to be identical with cathodic rays, and two sorts of non-deviable rays, one kind being very easily absorbed, the other resembling X rays and being very penetrating. Uranium emits principally the first kind, polonium gives only the second, and radium gives all three at once.

Let us now return to deviable rays; the material theory of Sir William Crookes and Mr. J. J. Thomson can be applied to them, and the consequences can be verified with the greatest facility. In a uniform magnetic field the trajectories perpendicular to the field are circumferential to the path ρ which leads the rays to the point of emission. For an oblique emission making an angle with the field, the trajectories are helices enveloping the cylinders with rays $\rho \sin \alpha$. By placing on a horizontal photographic plate parallel to the uniform field, a small lead box containing a few grains of radiferous barium forming a source of very small diameter, the rays are drawn down to the plate, and excite it on one side alone; a bundle of simple rays emitted in the plane normal to the plate and parallel to the field should show theoretically an arc of an ellipse of which the axes are in the proportion of 2 and π. The accompanying photograph shows these theoretical arcs, obtained by reversing the direction of the field, the one in air and the other *in vacuo*, on a photographic plate enveloped in black paper; the intensity of the magnetic field was about 4000 C.G.S. units.

If we do not enclose the photographic plate, and if we arrange on it several strips of paper or of metal to form screens, we observe in the print of the radio-activity dispersed by the magnetic field, a species of absorption spectra. Each trajectory has a different curvature corresponding to rays of different speeds and having different penetrating powers.

Here is an example of one of these prints, obtained in a field of about 1740 C.G.S. units; the screens are a strip of black paper, a strip of aluminium of 0·1 m.m. thickness, and a strip of platinum of 0·03 m.m. thickness. To obtain a pure spectrum so that at each point of the plate a bundle of rays are found, of which the trajectories have all the same curvature, the rays should be made to issue from the source so as to pass through a small round opening; the result is the same as the preceding one. This latter also shows a very intense impression, due to the secondary rays, provoked by the rays which were stopped by the lead cover over the active body, and in which was made a small opening through which the pure spectrum passed. The absorption varies with the distance of the screen from the active body, and the rays which are stopped by a screen placed on the plate are able to traverse this same screen when it is interposed at a point near their source.

These experiments leave little doubt as to the identity of the deviable rays with cathodic rays. However, it was necessary to prove that they carry charges of negative electricity, and that they are deviated by an electric field.

M. and Mdme. Curie, in a beautiful experiment, have shown that the rays of radium charge negatively the bodies that receive them, and that the source becomes charged positively. For this double experiment it is necessary that all the conductors and the source itself be completely enveloped in an insulating material, such as paraffin. For the active body examined the charge was $4 \cdot 10^{-13}$ C.G.S. units per square centimetre of radiating surface per second.

For my part, I have shown and measured the electrostatic deviation by projecting the deviated shadow of a screen placed perpendicular to the field, on a photographic plate. One of these apparatus is here shown, as well as one of the prints obtained in which on the two halves of the same plate appear the two deviated shadows corresponding to the reversal of the electric field, of which the intensity was $1 \cdot 02 \cdot 10^{13}$.

The balistic hypothesis attributes these phenomena to material masses transporting charges of negative electricity with very great rapidity. Let m be the material mass of a particle, e its charge, and v its velocity. We know that in a magnetic field of an intensity H, the radius of curvature ρ of the circular trajectory is given by the equation $H\rho = \frac{m}{e} v$. The numerical value of the product $H\rho$ serves to show the character of each simple ray. On the other hand, in an electric field of an intensity F, the parameter of the parabolic trajectory is $\frac{m}{e} \frac{v^2}{F}$. The knowledge of these two values gives $\frac{m}{e}$ and v. With a value of $H\rho = 1600$ I obtained approximately $v = 1 \cdot 6 \cdot 10^{10}$, and $\frac{e}{m} = 10^7$. These figures are entirely of the same order in value as those which led to the measurements made with cathodic rays, and the theoretical considerations with regard to Zeeman's experiment.

From the above figures we deduce that, from the fact of the deviable radio-activity under consideration, there escapes from each square centimetre of radio-active surface 1·2 m.grms. of matter in a thousand million years.

By extending these measurements to radiations of different and well-known natures, we ought to be able to determine if the relation $\frac{e}{m}$ is constant, or variable with one ray or another, and whether these do not differ only in their speeds; I have not yet finished the experiments I undertook to decide this fundamental question, but recently M. Kaufmann has attempted to elucidate the matter. He combined, at right angles, the magnetic and the electric actions; unfortunately, the experiment, which is very difficult to perform, did not give him one plate fit to measure. For the values of $H\rho$ comprised between 1800 and 4600, he found that the relation $\frac{e}{m}$ varied from $1{\cdot}3 \cdot 10^{7}$ to $0{\cdot}6.10^{7}$, and the speed v from $2{\cdot}3 \cdot 10^{10}$ to $2{\cdot}8 \cdot 10^{10}$.

The proof of a regular variation in the calculated relation $\frac{e}{m}$ is of considerable theoretical importance; if this relation was constant, as it seemed to be as the result of a large number of measurements, we might conclude that the slightly deviable rays, for which $H\rho$ is more than 5000, have speeds considerably greater than that of light.

On the other hand, theoretical considerations have given the idea that the speed could not surpass that of the propagation of electro-magnetic disturbances, that is to say, the speed of light, and we have been led to consider the mobile masses in a magnetic field as endowed with a particular inertia which is a function of the speed. Under these conditions the calculated mass ought to be apparent, or at least partly so, and it should increase indefinitely as the actual speed approaches that of light. The figures published by M. Kaufmann bear out this hypothesis.

Another consequence of this manner of looking at the question would be that there should be continuity between the deviable rays and those which are not, as the radius of curvature of the trajectories becomes infinite at the same time as the apparent mass.

The photographic print already mentioned, as well as one of the following ones, showed, on the contrary, a very distinct discontinuity, although in the second one the exposure was sufficiently prolonged for the impression of the least active rays, such as the penetrating non-deviable ones, to be distinctly visible.

This proof was obtained in the following manner:—In the uniform magnetic field of a permanent magnet, I placed, normally to the field, a photographic plate, then on this latter I arranged screens of lead fixed on a sheet of glass. These screens are pierced with openings normal to the plate, and destined to limit the width of the beam; in the path of these beams I arranged other screens, such as aluminium ones. Below the plate opposite a narrow slit in a strip of lead a small block of lead is placed having a deep cavity normal to the plate, and in which the radiant body is placed. We have thus a narrow, linear source normal to the plate and several millimetres in length. The cavity is covered with a thin sheet of aluminium to stop the light rays.

The figure represents a section made normally to the field of the beam, of which a part is deviated. Each beam corresponding to a determined speed gives an impression which is noticeably curved, as if the entire trajectory was marked on the plate. In these photographs the interior of the cylinders forming the screens is strongly affected by the secondary emission from the lead. The first picture shows that through each opening there passes an infinity of rays, constituting portions of the pure spectra. These meet with a strip of aluminium 0·1 m.m. in thickness, and traverse it without deviation, but not all with equal facility. The slightly deviated rays are penetrating, and excite secondary radiations when leaving the aluminium. The very deviable rays are stopped and give rise to points affected by an intense secondary radiation.

One only of the two categories of non-deviable rays appears in the form of two fine lines opposite the source; these are very penetrating rays, the others were arrested quite near the source.

Another picture shows the simple beam obtained by a double series of openings; by one of them we can sometimes pass two distinct trajectories.

The third figure is of interest, as it shows the straight beam traversing, without deviation, a sheet of aluminium placed obliquely to the line of trajectory; and, finally, the fourth one shows the transmission of simple rays through aluminium, and the secondary effects they produce.

The same method has enabled me to observe that the secondary rays were themselves deviated by the magnetic field, in the same way as the exciting rays.

The radiations from radium also comprise some which are very penetrating, consisting of the least deviable and the non-deviable rays, of which the properties seem to be the same as Röntgen rays. These penetrating rays are but very slightly absorbed, and consequently their action on a photographic plate or on the air is very feeble, so that, by the preceding methods, we can get no very exact idea of their intensity. If we interpose in their path a very absorbent screen, they traverse it partially, but at the same time they become partially transformed into more absorbable rays. This transformation recalls that of fluorescence, and, through the secondary action, the effect immediately behind the screen is stronger than if this latter was not there. The photographic plate receiving the radiations—filtered through a thickness of lead of 1 c.m.—gives a stronger impression through a sheet of lead of 1 m.m. thickness than in the uncovered regions. The diagram shows the effect of the radiations coming from the sides of a leaden box after having traversed 5 to 12 m.m. of the metal.

These secondary phenomena may partially account for the appearance of shadows given by the edges of all the transparent screens placed over the photographic plates.

All the facts I have just related have exclusively to do with the obscure radiations which traverse opaque bodies, such as metal, glass, mica, &c. But there exists also another, quite different, phenomenon, of which the effects are arrested by glass and mica; they are comparable to those produced by a vapour of a special nature. This phenomenon was discovered in 1899 by Mr. Rutherford and by M. and Mdme. Curie simultaneously.

Mr. Rutherford, while examining the radiations from thorium, observed that, besides the ordinary rays, there was another effect produced by an "emanation" consisting of a sort of vapour ionising the air. This vapour is deposited on bodies, principally those electrified negatively, and makes them momentarily radio-active. Mr. Rutherford made some very interesting measurements of this phenomenon.

At the same time, M. and Mdme. Curie discovered that, under the influence of radium, bodies became temporarily radio-active. This is not the secondary effect already described, but a persistent phenomenon which disappears comparatively slowly from the moment when the action of the radium ceases. M. Curie has called this "induced radio-activity," and has made a very complete examination of it. He has observed that the phenomenon is produced with great intensity in a closed space, that induced activity is the same on all bodies and practically independent of the pressure inside the enclosed space, but that the phenomenon is not produced if we maintain a complete vacuum by removing the gases produced; solutions of salts of radium produce the same effect with greater intensity than the solid salts. Liquids, water of crystallisation extracted from active salts, or the water separated from an active solution by a semi-permeable membrane of celluloid, remain strongly radio-active; it is the same with the gases. These excited bodies produce the same effects as radium; they emit a penetrating ray

which traverses the glass vessels which contain them and makes these latter luminous. Induced activity is gradually propagated in gases in a sealed tube, even through capillary tubes and imperceptible cracks; bodies are excited the more as the volume of gas is greater in proportion to their surface. Phosphorescent bodies become luminous when excited. In a recent work, MM. Elster and Geitel have observed that atmospheric air has properties analogous to those of excited gases, and they have been able to collect on wires negatively electrified, traces of radio-active products. The cause of this radio-activity is a problem of the greatest interest.

Finally, there is a remarkable method of induction, which is of such a nature as to demand the greatest reserve in the conclusions which might be formulated relative to the presence of new elements in radio-active bodies. Every inactive substance which has been added to a solution of a uranium or radium salt, and which has subsequently been removed by precipitation, has become radio-active, and loses this radio-activity very slowly. This fact was first observed by M. Curie and M. Giesel, who rendered bismuth radio-active in this manner. In the case of uranium, a trace of barium, precipitated in the form of sulphate, became notably more active than the uranium; barium thus excited emits only deviable rays, like uranium.

After this precipitation the uranium salt, brought back to the solid state, is less radio-active than before; this loss of radio-activity can even be accentuated by successive operations, but the products gradually and spontaneously regain their original activity. The temporary diminution of activity after solution is a general fact for salts of uranium and radium. With salts of actinium M. Debierne has communicated a very great activity to barium. The barium thus excited can be separated from inactive barium; it can be fractionated like radiferous chloride of barium, the most active portions being the least soluble in water and hydrochloric acid. M. Debierne in this manner obtained a product a thousand times more active than uranium. Barium thus excited behaves as a false radium, but it differs from true radium in the absence of the spectrum and in gradually losing its power with time.

Among the radio-active preparations a large number may be temporarily excited bodies. Such is the case with "polonium," which is apparently only excited bismuth.

Uranium and radium are characterised by their emission spectra and by the stability of their radio-activity. The spontaneous activity observed in the case of different salts after solution, might find an explanation in a phenomenon of auto-induction of the active molecules on the inactive one they are associated with.

The origin of the radiant energy of these radio-active bodies is still an enigma. By the material hypothesis it does not appear unreasonable, by applying the phenomenon of the evaporation of an odoriferous body, to compare the emanation to a sort of gas, of which the molecules would have masses of the same order of size as electrolytic ions, and to identify the radiations with the cathodic rays resulting from the dislocation of these ions, and causing at the same time the emission of X rays. We might thus ascribe the expenditure of energy to the dissipation of active matter. Although this hypothesis will account for most of the known facts, still there does not exist any precise experiment which sanctions it.

I must not, however, dwell longer on this subject, of which I have incompletely summarised the present position, by emphasising the physical part, which comes more especially within my province, although the chemical side has given rise to work of the greatest interest.

These questions have raised new hopes on the transformation of matter. Besides the exceptional conditions under which they enable us to examine the cathodic rays, they have raised, and continue to raise, fresh problems every day of which the first and most mysterious is the spontaneity of the radiations.

ACTION OF HYDROGEN ON SULPHIDE OF MERCURY.

By H. PÉLABON.

Crystallised sulphide of mercury, from which all the free sulphur it might contain has been removed, is attacked by hydrogen at a relatively low temperature. The reaction is quite distinct at 280°, but it is very slow.

Having placed some sulphide of mercury and hydrogen in a sealed tube, we found that, after heating to 280° for forty-five days, the gas, brought rapidly to the normal temperature of the laboratory, contained 17 per cent of sulphuretted hydrogen. We cannot be at all certain that this proportion would not have increased if we had continued the experiment.

For the purpose of examining the reduction of sulphide of mercury by hydrogen at temperatures above 280°, we used the method of rapid cooling which we described in our research on selenide of mercury (*Bull. Soc. Chim.*, Series 3, xxiii., p. 211).

Two distinct cases may be observed in the reduction of sulphide of mercury; it can be effected in closed tubes either in the presence of an excess of liquid mercury or in the absence of that metal.

1. *The Sulphide of Mercury is accompanied by an Excess of Mercury.*

At 360° we obtained the following results, ρ being the relation between the partial pressure of the sulphuretted hydrogen and the total pressure; this proportion being expressed in hundredths.

Time of heating. Hours.	Value of ρ.
5	10·69
24	26 61
48	50·12
74	73·51
94	78·78
144	79·30
342	78·11
480	78·50

The quantity of sulphuretted hydrogen formed at 360° thus tends towards a limit, characterised by the value 78·67 for ρ. This limit is reached after about ninety hours heating; its existence is due to the fact that the sulphuretted hydrogen and the mercury are able to react on each other under the same conditions of temperature. We placed some pure mercury and sulphuretted hydrogen in sealed tubes, and kept them at 360°. The results were:—

Time of heating. Hours.	Value of ρ.
6	91·39
15	79·92
192	79·29
215	78·91

Thus we again find a limit very near to that given by the direct action of the sulphide on hydrogen. It is as well to mention that the value of ρ does not depend either on the mass of the sulphide or the mass of the mercury, so long as these bodies are not entirely reduced to vapour at the temperature of the experiment. Neither has the pressure of the hydrogen gas any influence, as is distinctly shown by the following figures:—

Pressure of H at 0°. M.m.	Time of heating. Hours.	Value of ρ.
759	144	78·45
761	144	78·10
384	144	78·60
390	210	78·90
195	215	79·12

When the temperature increases, the limit value of ρ also increases. Thus, at 440°, we found:—

Time of heating. Hours.	Value of ρ.
2	44·48
14	85 25
24	85·18
43	85·41
144	85·20

Thus ρ reaches a limit of about 85·26 in a much shorter time than at 360°, as equilibrium may be considered as reached at the end of fourteen hours.

At 540° a value of about 92·10 was obtained for ρ. Finally, when the temperature is raised to above 540°, ρ has values which slightly pass the preceding figures. Thus, after four hours' heating, we found :—

At 585°	$\rho = 92{\cdot}13$
,, 590°	$\rho = 92{\cdot}00$
,, 610°	$\rho = 92{\cdot}81$

It is probable that, under these conditions of temperature, the method of rapid cooling does not give the exact composition of the gaseous mixture, the speed of the reaction becomes of the same importance as the speed of cooling. To sum up, we have—

At 360°	$\rho = 78{\cdot}67$
,, 440°	$\rho = 85{\cdot}29$
,, 540°	$\rho = 92{\cdot}10$

Thus the quantity of sulphuretted hydrogen goes on increasing with the temperature. This result is in complete accord with the law of the displacement of equilibrium by variations of temperature if we admit the following equation :—

$$HgS\ (solid) + H_2\ (gas) = H_2S\ (gas) + Hg\ (liquid),$$

which at 15° is accompanied by an absorption of heat, and is still endothermic at the temperature of our experiments.

2. *The Sulphide of Mercury is not accompanied by an Excess of Mercury.*

When we react with hydrogen and sulphide of mercury alone, two cases may occur; either the vapour-tension of the liberated mercury attains the maximum value or it remains lower than the tension of the saturating vapour.

In the first case, we have a recurrence of the system already examined; in the other, we have to study the equilibrium which is established between the following bodies :—Mercury vapour (the non-saturating vapour), solid sulphur, sulphur vapour (the saturating vapour), hydrogen, sulphydric acid.

In a general way, at a determined temperature and at an initial pressure of the hydrogen gas (also previously decided upon), the limit of proportion ρ is higher than the value ρ_1 of the same proportion which would be obtained when operating in the presence of an excess of mercury. The temperature remaining invariable, ρ diminishes as the pressure increases, but its value never gets lower than ρ_1.

At 360° we obtained the following figures :—

(*a*) The pressure of the hydrogen gas introduced into the tubes was about the normal pressure when measured at 0°.

Time of heating. Hours.	Pressure. M.m.	Value of ρ.
144	759	78·95
107	768	78·27
212	757	78·32

(*b*) The pressure of the hydrogen gas was about 380°.

157	387	78·75
225	390	78·11
212	389	78·63

(*c*) The pressure was about 190 m.m.

215	195	79·10
224	189	78·70
251	187	79·32

According to these results, the value of ρ would vary very little with the pressure; only the figures of the last series (*c*) were a little higher than the value $\rho_1 = 78{\cdot}67$ which we found in the first set of experiments.

Remarks.—It is easy to show that, starting with sulphide of mercury and hydrogen at a pressure of 760 m.m., we arrive at a system which should contain liquid mercury, provided the temperature is up to 360°. According to the equation of the reaction, the vapour-tension of the mercury set at liberty should be equal to the partial pressure of the hydrosulphuric gas, since the two bodies are formed simultaneously in equivalent proportions, as the H_2S represents two volumes of Hg.

The vapour-tension of the mercury should be therefore—

$$f = H \times \frac{\rho}{100}(1 + \alpha t) \quad . \quad . \quad . \quad . \quad (1),$$

α being the coefficient of dilatation of the gases at constant volume, t the centigrade temperature of the experiment, and H the pressure at 0° of the hydrogen gas originally introduced into the tube.

According to experiment, ρ has a value of about 78·6 when $t = 360°$. Thus, if $H = 760$ m.m., we have—

$$f = 760 \times 0{\cdot}786 \times (1 + 0{\cdot}00367 \times 360),$$

or—

$$f = 760 \times 1{\cdot}82.$$

But the tension of the saturating mercury vapour at 360° is about 760 m.m. $= F$; thus we get $f > F$, which cannot take place. A part of the mercury rapidly condenses to the liquid state, and we come back to the system examined before, and consequently we obtain the same limit for ρ.

The equation (1) enables us to calculate the pressure limit above which the mercury will be deposited, and below which it will remain in the state of a non-saturating vapour; it suffices to replace f by F, and ρ by the figure found in the first experiments; in this manner we get, if $F = 760$ m.m.,—

$$760 = H \times 0{\cdot}7867(1 + 0{\cdot}00367 \times 360),$$

from which we have—

$$H = 416 \text{ m.m.}$$

For pressures lower than 416 m.m., we ought to find different values for ρ from those obtained for pressures above this limit; this is exactly what happens if we refer to the results of the experiments made at pressures of about 190 m.m. At 440° we obtained the following results :—

(*a*) The pressure of the hydrogen gas is about normal.

Time of heating. Hours.	Pressure. M.m.	Value of ρ.
2	752	44·58
5	761	81·94
10	767	88·56
15	763	90·27
24	757	90·02
45	763	90·63
112	758	90·72

The equilibrium, reached after about fourteen hours, is characterised by a value for ρ of about 90·65.

(*b*) The pressure of the hydrogen was about 380 m.m.

2	375	68·36
8	382	94·06
12	387	94·51
18	377	94·23
48	384	94·36

The limit value of ρ is about 94·36.

(*c*) The pressure is about 190 m.m.

24	193	96·12
162	187	96·81
45	195	96·75
210	185	97·01

The average limit value of ρ is not more than 96·67. We made certain in every case that equilibrium was reached.

At 540° the pressure of the hydrogen was about the same as that of the atmosphere, and we obtained the following results:—

Time of heating. Hours.	Pressure. M.m.	Value of ρ.
½	761	97·19
14	756	97·84
72	763	97·00

The limit corresponds to $\rho = 97{\cdot}34$ (about).

For pressures below 760 m.m. the limit of ρ is above 97; it cannot, however, be measured with great accuracy, as the amount of the gaseous mixture to be analysed is very small—2 to 4 c.c. Finally, if the temperature exceeds 540°, ρ keeps a very nearly constant value of about 97·5.

Theoretical Considerations.—The figures found in the above experiments enable us to submit a relationship established by thermodynamics to the control of experiment—a relationship which has already been verified up to a certain limit by the figures obtained in the examination of the action of hydrogen on selenide of mercury.

If we write the equation of the reaction—

$$\underset{1}{HgS} + \underset{2}{H_2} = \underset{3}{H_2S} + \underset{4}{Hg},$$

applying the figures 1, 2, 3, 4 to the four bodies which take part in it, we can easily see that the general relation (1) which exists between the partial pressures of the gases and vapours contained in the system takes the form—

$$\log. \frac{p_1^{2v}\, p_2^{2v}}{p_3^{2v}\, p_4^{2v}} = F(T) \quad . \quad . \quad . \quad . \quad (2).$$

$2v$ is the volume occupied by the whole of the molecules of the simple and compound bodies. If the pressures are nil, we simply write—

$$\frac{p_1\, p_2}{p_3\, p_4} = f(T) \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (3).$$

Let us now see how this proportion will apply to each particular case examined.

1. In the reaction of hydrogen on sulphide of mercury in the presence of an excess of mercury, the pressure, p_4, of the mercury vapour is equal to the tension of the saturating vapour of this liquid. Let F be the tension, which is a function of the temperature only, the pressure, p_1, is also uniquely a function of the temperature for the same reason, since we operate with an excess of sulphur; we can therefore say—

$$\frac{p_2}{p_3} = \varkappa(T) \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (4)$$

or—

$$\rho_1 = \frac{100\, p_3}{p_2 + p_3} = \frac{100}{1 + \varkappa(T)}.$$

ρ_1 depends only on the temperature, and not on the pressure; this result is quite in accord with those obtained by experiment. We have seen, in fact, that no matter what the pressure was, we obtained, under the conditions indicated, $\rho_1 = 78{\cdot}67$ at 360°, $\rho_1 = 85{\cdot}26$ at 440°, &c.

2. If the hydrogen is made to react on sulphide of mercury alone we get—

$$p_1 = \phi(T) = P_1;$$

and, further, the equation of the reaction shows that at each instant we have—

$$p_3 = p_4 = P_3.$$

The proportion (3) then becomes—

$$\frac{P_2}{P_3^2} = \psi(T) \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (5).$$

The examination of this formula shows that the proportion of sulphuretted hydrogen gas in the mixture increases when the total pressure diminishes. The partial pressure of the hydrosulphuric gas and the total pressure, π, should, further, verify the following equations, which are deduced from the formula (5), $\pi = P_2 + P_3$:—

$$\frac{\pi - P_3}{P_3^2} = \frac{\pi' - P'_3}{P'^2_3} = \frac{\pi'' - P''_3}{P''^2_3} = \ldots = \psi(T) \quad . \quad . \quad (6).$$

We were able to determine, as has been shown, the corresponding values of ρ_2 for several different values of π. We repeat the values of this relationship, and place opposite those that the equations (6) enable us to calculate:—

Pressure of gas, π. M.m.	ρ_2 observed.	ρ_2 calculated.
758	90·65	—
386	94·36	94·73
187	96·67	96·84

The calculated figures are, as can be seen, slightly higher than those found by experiment; in spite of that, however, the concordance is fairly satisfactory.

Remarks.—When the system contains mercury in excess we can, in fact, write—

$$\frac{\phi(T)\, p_2}{p_3\, F} = f(T) \quad . \quad . \quad . \quad . \quad . \quad . \quad (7).$$

When, on the contrary, we make the hydrogen act on the sulphide alone, we have—

$$\frac{\phi(T)\, P_2}{P_3^2} = f(T),$$

from which we deduce—

$$\frac{P_2}{P_3^2} = \frac{p_2}{p_3\, F}.$$

F is given in tables of vapour tensions; it is equal to 2934 m.m. at 440°, $\frac{P_2}{P_3^2}$ can be calculated from the results of the experiments. We then find—

$$\rho_1 = 100 \frac{p_3}{p_2 + p_3} = 85{\cdot}61,$$

a figure which is very close to that found by experiment, which was $\rho_1 = 85{\cdot}26$.

The experimental verification of the relation (3) can be pushed further by examining the action of sulphuretted hydrogen on mercury.

Action of Sulphuretted Hydrogen on Mercury.

Sulphuretted hydrogen gas attacks mercury, giving a sulphide which can exist in the form of a non-saturating vapour; it is easy to see theoretically that, other things being equal, the limit value of ρ should be lower under these conditions than that obtained when the system contained an excess of sulphide of mercury. In fact, we get, according to the equation of the reaction,—

$$p_1 = p_2 = \omega_2, \qquad p_4 = F.$$

The proportion (3) is therefore written—

$$\frac{\omega_2^2}{\omega_3\, F} = f(T) \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (8).$$

ω_2 cannot be equal to the tension of the saturating vapour of the sulphide of mercury, without which we should again obtain the results of the first examination; now, this is not the case. Therefore, we necessarily have—

$$\omega_2 < \phi(T),$$

from which, allowing that we get—

$$\frac{\omega_2^2}{\omega_3\, F} = \frac{\phi(T)\, p_2}{p_3\, F},$$

we find the inequalities—

$$\frac{\omega_2}{\omega_3} > \frac{p_2}{p_3} \text{ and } \frac{\omega_3}{\omega_2 + \omega_3} < \frac{p_3}{p_2 + p_3}.$$

The pressure of the sulphuretted hydrogen gas being about 760 m.m. at 0°, we find at 440°—

$$\rho_3 = 100 \frac{\omega_3}{\omega_2 + \omega_3} = 80{\cdot}46.$$

The value of ρ_3 is therefore decidedly lower than the value $\rho_1 = 85{\cdot}26$.

The results of experiments at 440° are as follows, the pressure being about 760 m.m. :—

Time of heating. Hours.	Value of ρ.
2	95·00
15	82·80
24	81·86
72	80·14
96	80·57
112	80·67

At 540° we have already found that ρ_1 has a value slightly different to 92·10; by examining the action of sulphuretted hydrogen on mercury we found, with a pressure of about 760 m.m.,—

Time of heating.	Value of ρ.
5 minutes	97·11
10 ,,	88·48
15 ,,	84·65
25 ,,	84·27
12 hours	84·30

We thus again get distinctly $\rho_3 < \rho_1$. A quantitative verification could be made if we knew the tension of the saturating vapour of sulphide of mercury at each temperature.—*Bull. Soc. Chim.*, Series 3, xxv., No. 16.

ON THE

SULPHURIC AND DISULPHURIC ANHYDRIDES.

By G. ODDO.

The present ideas entertained regarding sulphuric anhydride are meagre and contradictory. According to Marignac (*Arch. Soc. Phys. Nat.*, 1833, vol. xxii., p. 255; 1875, vol. lii., p. 236) the two known modifications of this substance—the prismatic which melts at 18° and boils at 46°, and the fibrous, which is infusible, and changes into the prismatic form at 100°—are isomeric. According to Schultz-Sellak (*Bull. Soc. Chim.*, vol. xiv., p. 154; *Berichte*, vol. iii., p. 215), the second is the polymer of the former.

Buff (*Liebig's Ann.*, 4th Supplement, p. 127), and, later on, Weber (*Pogg. Ann.*, vol. clix., p. 313; *Berichte*, vol. xix., p. 3187), having shown that the prismatic product changes into the fibrous product only when it absorbs water, even a trace only, affirm that only a single modification of sulphuric anhydride exists, viz., the prismatic, fusible at 15°, of which the other is only a product of hydration. This opinion, attacked by Marignac (*Arch. Soc. Phys. Nat.*, [2], vol. lviii., p. 228), has been adopted by Rebs (*Bull. Soc. Chim.*, 1889, vol. i., p. 717; *Lieb. Ann. Ch.*, vol. ccxlvi., p. 356—382).

Finally, Ostwald, in his recent treatise on inorganic chemistry (*Grundlinien Anorg. Chemie*, p. 292) considers the two modifications as a case of polymorphism analogous to the two monochlorides of iodine. But still more regrettable than this diversity of opinion is the ignorance we must admit as to the identity or the difference of their chemical properties.

Wishing to attack this question, I have determined the molecular weights of the two modifications in solution in oxychloride of phosphorus; I found that the prismatic variety melts at 13·8°, and answers to the simple formula SO_3, the fibrous variety having the double formula S_2O_6. Further, I have noticed considerable differences in their chemical properties.

In the following lines I shall call the first modification sulphuric anhydride, and the second disulphuric anhydride.

Sulphuric Anhydride.

I prepared this anhydride by heating Kahlbaum's crystallised, fuming, sulphuric acid very slowly on the sand-bath, in a retort fitted with a ground glass stopper, to which was attached a small receptacle terminating with a point about 1 decimetre in length, plunged into the sulphuric acid. During the distillation this receptacle was kept in a water-bath at 30°, and when a desired quantity of the anhydride was collected, the two ends of the receptacle were fused before the blowpipe after removing it from the retort. I thus prepared two collectors each time, and these were kept constantly at 30° during the determinations, a temperature at which sulphuric anhydride is not polymerised.

As I have shown in a previous paper (*Reddiconti R. Accademia Lincei*, 1901, p. 453) oxychloride of phosphorus always contains products of hydration, which cannot be completely eliminated, either by distillation or crystallisation; further, there are always traces of moisture in the apparatus. To prevent all cause of error it is necessary to dehydrate everything. I succeeded in doing this very easily by using sulphuric anhydride itself in the following manner:—To a known quantity of the solvent contained in a cryoscope furnished with a stirrer, and carefully protected from the moisture of the atmosphere, I added one or more drops of the sulphuric anhydride from one of the collectors already prepared, but not weighed; it sometimes happens, if the oxychloride used contains too much moisture, that the first few drops cause an elevation of the point of congelation of the solvent; in this case we add a few more drops of sulphuric anhydride to obtain a considerable lowering of the congelation-point; I adopted this temperature as the congelation-point of the solvent, and I determined that from this point successive additions of anhydride caused lowerings of this point proportional to the concentration.

To make the determination, I use the anhydride from the second tube, which I weighed in a weighing bottle; by means of a file, I cut off the end of the tube; this I kept in the bottle, and poured the desired quantity of anhydride into the cryoscope. I then again sealed up the end of the tube, and made the cryoscopic determination. If in these various operations one is helped by a skilful assistant, almost every source of error can be guarded against. The following are the results obtained:—

First Determination.—The oxychloride crystallised at 3·928° by a Beckmann differential thermometer. After adding the unweighed sulphuric anhydride it crystallised at 3·458°; this figure was taken as the crystallising-point of the solvent. On adding the weighed quantity of sulphuric anhydride the point of congelation was found to be 1·362°.

By adopting the figure 69 (G. Oddo, *loc. cit.*) as the constant of the oxychloride of phosphorus we have:—

Concentration	2·2902
Lowering of the congelation-point ..	2·096°
Molecular weight	75·4

Second Determination.—The solvent crystallised at 3·812°, and after the addition of the unweighed sulphuric anhydride, at 2·310°. On then adding the weighed quantity of anhydride, we obtained for the point of congelation 0·488°. Therefore :—

Concentration	2·0643
Lowering of the congelation-point ..	1·822°
Molecular weight	78·1

For SO_3 the calculated molecular weight = 80·06.

Disulphuric Anhydride.

This was prepared by carefully distilling disulphuric acid on the sand-bath, and collecting the product at 10—13°;

it polymerises rapidly, but the polymerisation is not complete until after the lapse of several days.

The fibrous mass was cut, at the bottom of a deep mortar, with a pair of well-polished steel scissors, which are not attacked, and I kept the various pieces in weighing bottles; if they were all kept in the same bottle they would join together again and form one mass.

By means of platinum or even bone forceps, which are not attacked, the fragments can be easily transferred from the weighing bottle to the cryoscope.

In making the determinations I again dehydrated the oxychloride, and, at the same time, the apparatus, by dissolving unweighed pieces of the anhydride in the liquid, in sufficient quantity to cause a decided lowering of the congelation-point. The solution of this anhydride must be effected rather slowly, and it is necessary to keep on stirring.

The following are the results obtained:—

First Experiment.—The solvent crystallised at 4·212°, and after the addition of the unweighed fibrous anhydride at 2·329°, a temperature which I adopted as the congelation-point. By then adding the weighed quantity of fibrous anhydride the crystallisation took place at 2·145°.

Concentration..	1·9322
Lowering of the congelation-point ..	0·784°
Molecular weight	170·05

Second Experiment.—The following determinations were made in series, still the values obtained are sufficiently concordant; this proves the accuracy of the method.

The solvent crystallised at 4·898°, and after the addition of the unweighed fibrous anhydride at 4·569°.

Concentration.	Lowering of the congelation-point.	Molecular weight.
2·0360	0·843°	166·6
3·1505	1·347°	161·38
3·8400	1·682°	157·5

The calculated molecular weight for S_2O_6 is 160·12.

Third Experiment.—Finally, I will give a series of experiments made with the fibrous anhydride prepared some hours before the cryoscopic examination, but still having the appearance of a compact fibrous mass. The solvent crystallised at 3·625°, and after the addition of the unweighed fibrous anhydride at 3·155.°

Concentration.	Lowering of the congelation-point.	Molecular weight.
1·9893	1·119°	122·66
4·0609	2·365°	118·47
4·4510	2·543°	120·77

Differential Characteristics between Sulphuric Anhydride and Disulphuric Anhydride.

These anhydrides, besides their already known physical properties, differ in several chemical points. I will here only mention the following facts.

First of all, as a dehydrating material sulphuric anhydride is much more active than disulphuric anhydride. If, for example, we let a drop of sulphuric anhydride fall on an organic tissue, the latter is immediately carbonised; in the case of animals, very deep ulcers are formed; on the other hand, disulphuric anhydride does not affect organic tissues, that is if the contact is not sufficiently prolonged as to allow of hydration from the atmosphere. I have squeezed this anhydride between my fingers without suffering the least inconvenience. A fact already observed by Schulz-Sellak, is that sulphuric anhydride dissolves immediately in sulphuric acid; on the other hand, disulphuric anhydride dissolves very slowly. I have observed further that if the sulphuric acid has been well-cooled, when pieces of disulphuric anhydride and a few pieces of ice are thrown into the acid together, no energetic action takes place; the anhydride dissolves slowly, and only after several minutes, but with continual stirring we eventually get complete solution.

The same thing occurs with organic substances, if we take care to avoid all sudden increases of temperature which, causing the rapid dissociation of the disulphuric anhydride into sulphuric anhydride, would cause the effects of the latter to be attributed to the former.

To cite one example only, sulphuric anhydride reacts energetically and very quickly on powdered camphor, which is partly carbonised, and partly transformed into a liquid body; on the other hand, by making an intimate mixture at the ordinary temperature of disulphuric anhydride and powdered camphor, nothing in particular is observed, and it is only after having triturated the mixture for some time that the reaction takes place; but as soon as it commences, the disengagement of heat is so great that it causes the instantaneous dissociation of all the disulphuric anhydride into sulphuric anhydride. The reaction then takes place rapidly, as in the case when sulphuric anhydride was used, and the final result is the same. I will also mention a few new facts concerning these two anhydrides, which will show how necessary and interesting the thorough examination of these substances is, a research which I propose to continue.

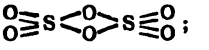
Sulphuric anhydride does not react on the metals; if a few drops of this anhydride, poured into a test-tube, are placed in contact with various metals, we observe that sodium and potassium float thereon, and the other metals, such as Mg, Zn, Cu, Sn, Pb, Ni, and Cd, all in the form of powder, and Hg, &c., sink without any reaction taking place.

In the same manner the oxides of the metal do not react with sulphuric anhydride at the ordinary temperature; I have tried HgO, SnO, CuO, ZnO, PbO_2, BaO, and Na_2O always with negative results.

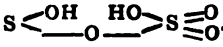
Disulphuric anhydride, at the bottom of a test-tube exposed to the air, remains almost unchanged for several days; a part of it sublimes and reaches a certain height, where a white cloud is formed which, in spite of agitation, will not rise further, and protects the anhydride remaining underneath from the action of the moisture in the atmosphere. Sulphuric anhydride left at the bottom of a test-tube is soon transformed into disulphuric anhydride.

While postponing theoretical considerations to a future and more complete paper I will here remark that if we consider the atom of sulphur as a hexad, the formula for sulphuric anhydride will be:—

and that of disulphuric anhydride:—



the first term of hydration of this latter is disulphuric acid—



Further, if we admit that the atoms of oxygen have the form of an elongated ellipsoid and are disposed symmetrically round the atom of sulphur, as if on the sides of an equilateral triangle of which the sulphur occupies the centre, we can easily see why sulphuric anhydride is stable above 27° and polymerises below that point; the three atoms of oxygen, in their vibrations at a low temperature, move in the direction of their extremities; this results in a tension which, by analogy with the trimethylenic compounds, may be called external; it is for this reason that any disturbing cause, such as traces of moisture, suffices to turn round the atom of oxygen, with the formation of a radicle which unites with another identical radicle giving a molecule of disulphuric anhydride. The opposite happens when a high temperature acts on this latter anhydride; and for this reason it is dissociated into two molecules of sulphuric anhydride.—*Bull. Soc. Chim.*, Series 3, vol. xxv., No. 20.

A NEW INVESTIGATION CONCERNING THE ATOMIC WEIGHT OF URANIUM.*

By THEODORE WILLIAM RICHARDS
and
BENJAMIN SHORES MERIGOLD.†

Introduction.

Our knowledge of uranium dates from the year 1789, when it was first recognised as an element by Klaproth. It can by no means, therefore, be classed with the new elements, nor is it of great rarity. Nevertheless, comparatively few determinations of the atomic weight of this element have been made, and of these, one only has been carried out with the degree of accuracy necessary in work of this kind. During the fifty years following the discovery of uranium a number of atomic weight determinations were made by Berzelius, Arfvedson, Schönberg, Marchand, and Rammelsberg. This early work is now of historical interest only, for the results vary widely, and in some cases are of such a nature as scarcely to be considered quantitative, in the modern sense of the word. For example, Rammelsberg obtained results varying from 184 to 234, calculated upon the modern basis.

In 1841 Péligot discovered that the substance then known as uranium was not an element, but an oxide. This discovery, while it did not impair the value of the analytical work previously done, necessitated a re-calculation of the numerical value of the atomic weight. The new value was 120, and this remained practically unchanged during the next thirty years. When the periodic classification of the elements was first suggested, uranium, with the atomic weight 120, was one of the elements for which there was no place. From a study of the properties of uranium and its compounds, Mendeléeff declared that the atomic weight of uranium was probably 240 instead of 120 (*Annalen der Chemie u. Pharmacie*, Supp. Vol. viii., 178 *et seq.*). The question was not definitely settled until Zimmermann, in 1885, carried out the suggestions of Mendeléeff, and by specific heat and vapour density determinations confirmed the higher value (*Annalen der Chemie u. Pharmacie*, ccxvi., 1).

Owing to the wide variations in the published results, the atomic weight of uranium has long been considered one of the least satisfactorily determined of the atomic weight values. A glance at the results thus far obtained is sufficient to show the need for further work in this line. A complete *résumé* of the older work upon the subject is to be found in Clarke's recent work on the atomic weights ("A Re-calculation of the Atomic Weights," by F. W. Clarke, Smithson. Misc. Coll., "Constants of Nature," 1897, Part V., 263). The following table summarises those investigations which seem to possess even a little quantitative value:—

Less Inaccurate Previous Work on the Atomic Weight of Uranium.

O = 16·000.

1841.	Péligot (*Compt. Rend.*, xii., 735; *Ann. Chim. Phys.*, 1842, [3], v., 5).—Analysis of green chloride	240·0±
1842.	Ebelmen (*J. Prkt. Chem.*, 1842, xxvii., 385).—Combustion of oxalate	238·0±
1843.	Wertheim (*J. Prkt. Chem.*, 1843, xxix., 209).—Double acetate of sodium and uranium	239·0±
1846.	Péligot (*Compt. Rend.*, 1846, xxii., 487).—Combustion of oxalate and acetate ..	240·0±
1886.	Zimmermann (*Ann. d. Chem.*, 1886, ccxxxii., 299).—Reduction of oxide, U_3O_8 to UO_2	239·6
1886.	Zimmermann (*Ann. d. Chem.*, 1886, ccxxxii., 299).—Ignition of double acetate	239·5

The work of Ebelmen, Wertheim, and the early work of Péligot is necessarily of little weight in assigning a probable value to the atomic weight of uranium. In some cases the material used was impure, and in others the methods of analysis were faulty. Consequently, it is not surprising to find differences of whole units in the individual determinations of each series.

Péligot's later determinations from the oxalate is perhaps the best of the early work. His material was carefully purified, and his method is far preferable to the work of Ebelmen and Wertheim. By combustion analysis he determined the ratio between uranium oxide and carbon dioxide. Thus he eliminated the error involved in weighing a crystallised salt which would probably contain more or less included water. The principal sources of error are the questionable use of combustion analysis in atomic weight investigations, and the possibility of unoxidised carbon remaining in the uranium oxide. His best results vary from 239·4 to 241·1.

The work of these chemists, though a great improvement over the attempts of Rammelsberg and the other early workers, leaves much to be desired, and does little more than give an approximate idea of the probable value of the atomic weight of uranium.

Zimmermann's investigation of the ratio between the oxides UO_2 and U_3O_8 was much more carefully carried out, and is the only work thus far published that is worthy of serious consideration. Using carefully purified material, and giving attention to detail, Zimmermann oxidised the lower oxide by means of a stream of oxygen, and observed the gain in weight. His results for the atomic weight varied from 239·49 to 239·76, an extreme difference of 0·27, or 0·11 per cent. The average was about 239·6. The chief probable cause of error in this method is the difficulty which is always experienced in forming a more voluminous solid from a less voluminous one. Uranous oxide has a specific gravity of 10·2 while the "uranoso-uranic" oxide has a specific gravity of only 7·3. The great increase of volume which occurs when the higher oxide is formed must tend to protect particles of the lower oxide from the action of the oxygen. Hence the gain in weight will be too small, and the apparent atomic weight of the metal too large (compare Richards and Baxter, *Proc. Amer. Acad. Arts Sci.*, 1898, xxxiv., 351; *Ztsch. Anorg. Chem.*, 1899, xxi., 251). It is clear that a very small deficiency in the weight of the higher oxide must cause a great increase in the apparent atomic weight.

Moreover, any incompleteness in the reduction by which the lower oxide was prepared, or any retention or occlusion of gases within this oxide, would also tend to raise the apparent atomic weight. Hence one is inclined to believe, even without further evidence, that Zimmermann's result for uranium must be too high.

A new determination of the atomic weight of uranium has recently been made by J. Aloy (*Comptes Rendus*, 1901, cxxxii., 551).* The method employed differs materially from any previously used in uranium work. The values obtained are derived from the ratio between the weight of nitrogen and that of uranous oxide contained in crystallised uranyl nitrate. Uranyl nitrate was purified by repeated crystallisation. A quantity of the pure nitrate, the weight of which need not be known, was put into a boat, and the boat surrounded by a section of platinum tube, to prevent loss of material. The whole was placed in a combustion tube between spirals of reduced copper. One end of the combustion tube was connected with a carbon dioxide generator, and the other with an absorption apparatus containing a concentrated solution of potash.

After sweeping the air out of the apparatus with a current of carbon dioxide, the nitrate was heated so long as evolution of nitrogen occurred, the temperature being finally raised to red heat. The reduced copper was kept at red heat throughout the operation. When it was certain that no more nitrogen was evolved, the green oxide remaining in the boat was reduced by hydrogen to uranous

* Contributions from the Chemical Laboratory of Harvard College. From the *Proceedings of the American Academy of Arts and Sciences*, xxxvii., No. 14.

† The greater part of the work described in this paper was presented to the Faculty of Arts and Sciences of Harvard University by B. S. Merigold, as a thesis for the degree of Ph.D., in June, 1901.

* This work is discussed rather fully here, since it is too recent to have been included in Clarke's book.

oxide and weighed. The nitrogen was transferred to a measuring tube reading to tenths of a cubic centimetre. From the ratio of the weight of this volume of nitrogen to the weight of the oxide, the atomic weight is calculated.

The following are the results of the eight determinations given:—

Atomic Weight of Uranium.

N = 14·04.

Volume of nitrogen. C.c.	Atomic wt. of uranium.
15·25	239·3
33·5	239·4
38·0	239·6
52·5	239·5
81·25	239·4
125·0	239·5
151·2	239·4
165·0	239·4
Average	239·4

This method has the merit of simplicity, and does not involve the weight of the crystallised salt. There are, however, several sources of possible constant error that have not been taken into account. When crystallised uranium nitrate is heated, it first melts in its water of crystallisation. As in all similar cases, it requires the very greatest care to prevent spattering while the crystal water is being driven off. It was undoubtedly as a precaution against loss of material in this way that Aloy used his platinum tube. By the time the crystal water is expelled, the fused mass has hardened into a solid cake, changing in colour from yellow to orange, and finally to the green of urano-uranic oxide, U_3O_8.

This method of preparing the green oxide from pure uranyl nitrate was used in the work to be described in the following pages. It was *invariably* found, however, that during the decomposition of the dried nitrate, and the subsequent oxidation, the oxide first produced forms a protecting crust, as it were. This prevents, or at least very materially retards, the decomposition of the material within the interior, *even when the temperature is maintained for several hours at red heat*. On the outside, the material had the appearance of being completely converted to oxide. On powdering the lumps, however, and again heating, there was *in every case* a further evolution of nitric fumes. Moreover, nitrogen itself is often retained by oxides prepared in this way (Richards and Rogers, *Proc. Amer. Acad. Arts Sci.*, 1893, xxviii., 200; also Richards, *Proc. Amer. Acad. Arts Sci.*, 1898, xxxiii., 399). It seems thus extremely probable that the quantities of nitrogen measured by Aloy were in every case too small. Obviously, until this point is definitely settled, Aloy's results must be regarded with more or less suspicion.

It has been pointed out that reduction is usually much more complete than oxidation (Richards and Baxter, *loc. cit.*). During the reduction of an oxide, there is formed, perhaps, by the removal of a portion of the oxygen, a kind of skeleton framework, giving to the remaining substance a porous structure which enables the reducing gas to penetrate farther into the interior of the mass, until reduction is complete. Owing to this action, it is probable that when the green oxide of uranium is finally reduced by hydrogen, all the nitrogen is expelled, and the final product is pure uranous oxide. Consequently, the weight of uranous oxide used in the calculation is probably nearly correct, the principal error being in the volume of nitrogen.

Aside from this special objection to the use of this method in its application to uranium, there is the general objection to the use of such a method where great accuracy is desired. The exact measurement of small quantities of gas offers considerable opportunity for error, especially when, as in this case, the gas is first to be transferred from the collecting to the measuring apparatus. When the volume or weight of a gas is involved in an atomic weight investigation, it is customary to work with as large volumes as possible, thus reducing to a minimum the effect of the errors inevitably connected with the measurement of the gas. The exact measurement of a volume no larger than 165 c.c., even—the largest volume measured by Aloy—is a matter of considerable experimental difficulty, while with the smaller volumes, 15, 33, and 38 c.c., errors of at least 0·1 per cent are not only possible, but extremely probable. A difference of one-tenth of 1 per cent in the volume of nitrogen makes a difference of 0·3 in the value of the atomic weight. The errors of collection and transference of the gas are more likely to result in reading too small rather than too large volumes, giving too high values for the atomic weight.

From these considerations, it is evident that Aloy's results are at least somewhat doubtful. Aloy gives notice of his intention to apply this method to the determination of other atomic weights, but it is to be hoped that before doing so he will clear up some of the doubtful points in connection with the process. As carried out in this investigation, the method certainly is not a valuable addition to the methods of atomic weight determination.

From the earlier results Clarke computed the value 239·6, while the German Committee recommend 239·5. Both figures are practically identical with Zimmermann's figures.

The investigation herein described was undertaken with the hope that by increasing the experimental basis of our knowledge of the subject, we might be able to reduce to somewhat narrower limits our present uncertainty in regard to the real value of this constant.

(To be continued).

NOTICES OF BOOKS.

Assaying and Metallurgical Analysis, for the Use of Students, Chemists, and Assayers. By E. L. Rhead, Lecturer on Metallurgy, Municipal School of Technology, Manchester, and A. Humboldt Sexton, F.I.C., F.C.S., Professor of Metallurgy, Glasgow and West of Scotland Technical College. London, New York, and Bombay: Longmans, Green, and Co. 1902. Pp. 431.

The present volume was written with the intention of providing the student, chemist, or assayer with a handbook sufficiently comprehensive to include the greater part of the work likely to be required in the laboratory or assay office. That the authors have achieved their object will be evident to anyone who looks through the book.

A work such as this is not intended so much for the mere student who is actually learning the subjects treated, but it is of great service to the assayer in general as a book of reference and laboratory companion.

The book is divided into three parts. Part I. deals with laboratory appliances and general processes, such as sampling, weighing, the management of furnaces, the reagents used in both dry and wet assaying, &c.

In Part II., which is the most important part of the volume, the general plan followed has been to consider under the name of each metal, (*a*), the materials and products in which its estimation is commonly required, accompanied by a short description of the same to make their identification easy; (*b*), the dry tests for, and the important chemical reactions of the metal, with, in some cases, special instructions for the detection of small quantities; (*c*), the methods of conducting the dry assay by fusion or otherwise, followed by applications to special materials where modifications are necessary; (*d*), methods of preparing solutions for the estimation of the metal; and (*e*), methods for gravimetric, volumetric, and colorimetric estimations.

Part III. is on metallurgical analysis, and commences with the analysis of alloys of all kinds, and the commoner metals, such as copper, zinc, tin, &c., in their commercial forms; this is followed by the analysis of iron ores, iron and steel, ferro-aluminium, chromium, tungsten, &c., slags and refractory materials; and, finally, the examination of coal, coke, and gaseous fuels.

There are five appendices, containing useful tables and special tests, such as the cyaniding tests for gold, tables for qualitative analysis, &c.

The book is well printed and bound, and the illustrations, though not numerous after the first part is concluded, are good, while the table of contents and the index are efficient and well carried out.

CORRESPONDENCE.

ANALYSIS OF TIN ORES.

To the Editor of the Chemical News.

SIR,—I read with interest the translation of J. A. Muller's paper on the above subject (*Bull. Soc. Chim.*, Series 3, xxv., No. 23) which appeared in the CHEMICAL NEWS of March 27th (vol. lxxxv., p. 147), and was pleased to find that the author's results fully confirm the reliability of the hydrogen-reduction method of assaying ordinary tin ores.

I have used the process for over thirty years, and do not hesitate to recommend it for general adoption in laboratories, as being far and away the quickest, the simplest, and the most accurate.

It is only fair to here mention that the first chemist to suggest the use of hydrogen in the analysis of tin ores was Dr. William McCowan, F.I.C., &c., who, in 1871, when we were brother students under Professor Arnulf Schertal, perfected the process which is now credited to other analysts.—I am, &c.,

R. W. EMERSON MACIVOR.

The Laboratory,
29, Fenchurch Street, E.C.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. CXXXIV., No. 11, March 17, 1902.

Heat developed during Reactions between Bodies in the Solid and Gaseous State.—M. Ponsot.—Two bodies, A and B, at a temperature T, are allowed to interact, giving rise to other bodies, for example M and N. The author examines such a reaction, choosing suitable bodies. Two systems are formed, one consisting of the bodies A and B, the other of the bodies M and N, in each case the solid bodies being accompanied by a mixture of their vapours. He finds that the heat brought into play by the total reaction of one body in a solid state, and supporting a pressure produced by the tension of the vapours it emits and the heat emitted during the total reaction, and under the constant volume of the same bodies when in the gaseous state, tends towards the same value at absolute zero.

Heat of Solution of Solid and Liquid Ammonia at about −75°, and Latent Heat of Fusion of Solid Ammonia.—G. Massol.—The latent heat of fusion of ammonia has never been determined experimentally, but this constant is necessary if the heats of formation of the ammonia salts are to be compared with those of other alkaline salts, all the bodies being taken under the same initial conditions. The method adopted by the author for finding this constant consists in dissolving in water in a calorimeter (1) liquid ammonia at −75°., *i.e.*, at the temperature of solidification, (2) solid ammonia taken at a temperature as near as possible to −75°, the fusion temperature. The algebraic difference between the numbers obtained from these two thermic reactions gives the required constant, with a slight error, on account of the impossibility of obtaining the body in the two states exactly at the same temperature. The mean value obtained is −0·068 cal. for solid ammonia and +1·77 cal. for liquid ammonia. From these the number −1·838 cal., the molecular latent heat of fusion, can be obtained.

Volumetric Estimation of Thallium.—V. Thomas.—The author employs a modification of Feit's method for estimating thallium volumetrically. A thallic salt is treated with potassium iodide, and a greenish black precipitate is obtained. This may be considered as a mixture of thallous iodide and iodine, resulting from the decomposition of an unstable triiodide, $TlI_2 = TlI + I_2$. To estimate thallium volumetrically the solution should contain the metal in the form of thallic sulphate, and the transformation into thallic sulphate is absolutely necessary in the case of thallium existing in the liquid in the form of a halogen salt. Excess of potassium iodide is added, and enough arsenious solution to produce a pure yellow precipitate of thallous iodide. This is filtered, and to a portion of the filtrate excess of arsenious acid is added.

Acid and Basic Sulphates of Neodymium and Praseodymium.—Camille Matignon.—The author announces the existence of the new salts $Nd(SO_4H)_3$, $Pr(SO_4H)_3$, $(NdO)_2SO_4$, $(PrO)_2SO_4$, and examines certain of their properties.

Alkalimetric Estimation of Disodic Methylarsinate or Arrhenal.—A. Astruc.—The author finds a simple and rapid method of estimating disodic methylarsinate, based on its alkaline reaction. The methylarsinate when analysed is found to correspond to the formula—

$$AsO{-}ONa,5H_2O,$$

with CH_3 and ONa attached to As,

the theoretical percentage of water being 32·84 and of arsenic 27·37. A standard solution of this substance is titrated against a half decinormal solution of sulphuric acid, and the results obtained are satisfactory.

Some Derivatives of Arabinose.—G. Chavanne.—The author examines a sugar containing five carbon atoms, arabinose. This he prepares quite pure and in considerable quantity in order to obtain its oxidation products. By employing Kœnig and Knorr's method he obtains an acetochlorarabinose and an acetobromoarabinose in a pure state.

The Supposed Binaphthaleneglycol.—R. Fosse.—In a preceding research the author showed that several bodies formed during the action of chloroform on the β-naphthol did not possess the constitution attributed to them. He now shows that the substance known under the name of binaphthalene glycol, with constitution—

$$C_{10}H_6{-}C{-}OH$$
$$\quad\quad\quad

plying the rule of mixtures for systems in solution, the molecular refraction may be expressed as follows:—

ref. RNa—ref. RH = ref. NaOH—ref. H_2O = K (constant).

The value of this constant is comprised between 1·5 and 1·6. Such reasoning necessarily supposes that the radicle R has the same structure in the acid and in the soda-salt. If this is not the case values for K differing from 1·55 should be obtained. The author establishes values for K between 3·5 and 4 for a certain number of isonitroso-compounds of the fatty series, oximidocyanacetic and isonitrosomalonic ethers, isonitrosoacetone, isonitrosocamphor, &c. Such belong to the category of substances which Hantzsch calls the pseudo-acids, substances whose constitution varies when they pass to the state of the neutral salts. The author intends to publish details of his research on this subject.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xxv., Nos. 16—17.

Action of Hydrogen on Sulphide of Mercury.—H. Pélabon.—(See p. 172).

Action of Mercuric Oxide on Aqueous Solutions of Metallic Salts.—A. Mailhe.—Already noticed.

A New Crystallised Salicylate of Bismuth. — P. Thibault.—Already inserted in full.

Acidimetry of Phosphoric Acid by means of Baryta Water.—J. Cavalier.—Already inserted in full.

Action of Fuming Nitric Acid on Dimethylacrylate of Ethyl.—L. Bouveault and A. Wahl.—When dimethylacrylate of ethyl is poured into fuming nitric acid the mixture becomes strongly heated, and should be kept cool; on adding water a heavy oily liquid separates out, this is nitrodimethylacrylate of ethyl. The author describes in full the details of its preparation. When purified by re-distillation it consists of a very bright yellow liquid with a disagreeable odour. It is insoluble in water and dilute acids, insoluble in cold caustic alkalis; it dissolves, however, on heating or after prolonged contact in the cold, but not without decomposition. Its potassium salt, in which a molecule of hydrogen is replaced by one of potassium, gives coloured precipitates with the metallic salts.

Constitution of the α- and β-Nitrodimethylacrylic Ethers.—L. Bouveault and A. Wahl.—A long paper not suitable for abstraction.

Action of Bromine on Carvacrol in the presence of Bromide of Aluminium.—F. Bodroux.—Five grms. of carvacrol are poured, drop by drop, into 100 grms. of cold bromine containing 1 per cent of aluminium in solution. The reaction is energetic, and torrents of hydrobromic acid are given off. After six hours the excess of bromine was driven off by evaporation, and, as a result of the operation, a solid white mass was obtained, impregnated with a gummy substance, which, however, could be removed by washing with a little cold alcohol. This white product is soluble in warm dilute alcohol, and more so in chloroform, and on cooling deposits long, silky, white needles, fusible without decomposition at 208°. Its physical properties and its analysis show that the body obtained is tetrabromorthocresol—

$$C_6Br_4<^{CH_3\ (1)}_{OH\ (2)}.$$

Some Iodised, Etherial, Phenylic Derivatives.—P. Brenans.—Already noticed.

Examination of a New Class of Organic Compounds; Cyclic Isonitriles and Nitriles.—A. Sabanejeff and M. Prosin.—After describing the method of preparing these new compounds, the authors conclude that these isonitriles and nitriles are cyclic compounds of which the ring consists of several atoms of carbon and nitrogen in which the hydrocarbon group could easily pass from the nitrogen to the carbon, and *vice versâ*. The isonitriles and nitriles at present known represent the most simple case only, and are in the same relation to the cyclic isonitriles that ethylene is to the polymethylenic hydrocarbides.

The Constitution of Licareol (Linalol).—Ph. Barbier.—For various reasons which the author fully explains in this paper, he proposes to replace the tertiary formula—

$$CH_3—\underset{\displaystyle CH_3}{\overset{|}{C}}=CH—CH_3—CH_2—\underset{\displaystyle CH_3}{\overset{|}{C}}(OH)—CH=CH_2,$$

which has been attributed to licareol, but which is really myrcenol, by the primary formula—

$$CH_3—\underset{\displaystyle CH_3}{\overset{|}{C}}=CH—CH_2—CH_2—\underset{\displaystyle CH_3}{\overset{|}{C}}=CH—CH_2OH,$$

which is common to both licareol and lemonol, and to consider these two alcohols as stereo-chemical isomers.

MISCELLANEOUS.

Royal Institution.—A General Monthly Meeting of the Members of the Royal Institution was held on the 7th inst., Sir James Crichton-Browne, Treasurer and Vice-President, in the Chair. Messrs. W. A. Bailey, J. M. Barr, C. W. Darley, W. E. L. Gaine, Carl Hentschel, J. C. Inglis, G. Northcroft, H. C. Plimmer, F. E. Robertson, and G. A. Wilson were elected Members. The special thanks of the Members were returned to Dr. Francis Elgar, F.R.S., for a donation of £50 to the Fund for the Promotion of Experimental Research at Low Temperatures.

Hydrated Thiocyanate of Cobalt.—H. S. Kournakof.—By slowly evaporating a solution of a mixture of equivalent parts of $CoSO_4$ and $Ba(CNS)_2$, we obtain the hydrate $Co(CNS)_2\ 3H_2O$, crystallised in large, almost black plates belonging to the rhombic system, and which transmit a reddish-violet light through a very slight thickness. This hydrate effloresces in the air, and gives a yellowish-brown anhydrous salt. This latter is obtained crystallised in granular crusts by evaporating the solution on the water-bath. No salts are obtained with colours intermediate between violet and yellow, which seems to indicate that there is no intermediate hydrate.—*Journ. Soc. Phys. Chim. R.*, vol. xxxii., p. 354.

MEETINGS FOR THE WEEK.

Monday, 14th.—Society of Arts, 8. (Cantor Lectures). "Glass for Optical Instruments," by Richard T. Glazebrook, M.A., D.Sc., F.R.S.

Tuesday, 15th.—Royal Institution, 3. "Recent Methods and Results in Biological Inquiry," by Allen Macfadyen, M.D., B.Sc., Fullerian Professor of Physiology, R.I.

Wednesday, 16th.—Microscopical, 7.30. Exhibition of Pond Life.

—— Society of Arts, 8. "Photography as Applied to Architectural Measurement and Surveying," by J. Bridges Lee, M.A.

Thursday, 17th.—Royal Institution, 3. "The Oxygen Group of Elements," by Prof. Dewar, M.A., LL.D., D.Sc., F.R.S., Fullerian Professor of Chemistry, R.I.

—— Society of Arts, 4.30. "Recent Developments in Punjab Irrigation," by Sidney Preston, C.I.E.

—— Chemical, 8. "Oxonium Salts of Fluoran and its Derivatives," by J. T. Hewitt and J. N. Tervet. "The Influence of certain Acidic Oxides on the Specific Rotations of Lactic Acid and Potassium Lactate," by G. G. Henderson and D. Prentice. "The Amounts of Nitrogen as Ammonia and as Nitric Acid, and as Chlorine in the Rain-water collected at Rothamsted" and "The Amounts of Nitrogen as Nitrates and Chlorine in the Drainage through Uncropped and Unmanured Land," by N. H. J. Miller.

Friday, 18th.—Royal Institution, 9. "The Autocar," by Sir John H. A. Macdonald, K.C.B., LL.D., F.R.S., M.Inst.E.E.

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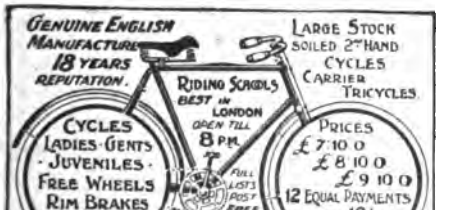

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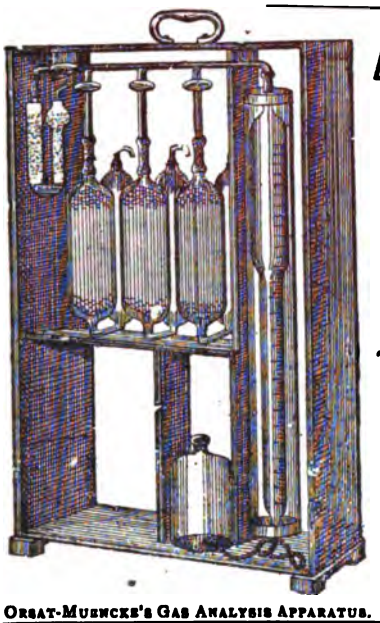

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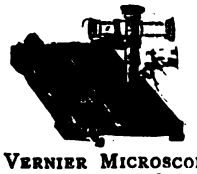

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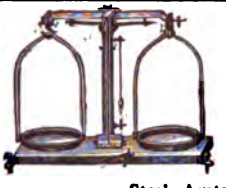

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THE CHEMICAL NEWS.

VOL. LXXXV., No. 2212.

THE COMPOSITION OF THE CANARY-YELLOW ARSENITE OF SILVER.

By J. ALFRED WANKLYN,
Corresponding Member of the Royal Bavarian Academy of Sciences.

THE canary-yellow arsenite of silver which every schoolboy knows, and which is described in chemical text-books as "the tetrargentic salt, $2Ag_2O.As_2O_3$," has been recently analysed in my laboratory. The result of the analysis has astonished me, and is certainly interesting.

The following are the details and the figures of the analysis:—

The arsenite of silver, as is well known, is produced when the gas from the Marsh's apparatus is led into solution of nitrate of silver. It separates from the filtered solution (the reduced silver having been left on the filter-paper) on the cautious addition of dilute ammonia to the filtered solution.

A quantity of the arsenite of silver prepared in that manner was washed and dried in the water-bath and analysed.

1·0135 grms. gave 0·966 grm. of AgCl and 0·277 grm. of As_2S_3. The formula $3Ag_2O.As_2O_3$ requires—

	Calculated.		Found.
Ag_6	648	72·48	71·73
As_2	150	16·78	16·66
O_6	96	10·74	—
	894	100·00	

The formula was confirmed by a synthetical experiment. A weighed quantity of As_2O_3 was dissolved in water, and an insufficient quantity of ammonia added, and then the observation was made that, on mixing with this solution a known quantity of nitrate of silver (excess of nitrate of silver being taken), the solution became powerfully acid during the precipitation of the yellow arsenite of silver. Dilute ammonia was then carefully added so as just to saturate the acid, and the resulting canary-yellow arsenite of silver was separated by filtration, washed, slightly pressed, dried at 212° F., and weighed. The theoretical figures were experimentally obtained.

It is noteworthy that, in this experiment, the yellow colour of the precipitate was *not* lost on drying.

The Laboratory, New Malden, Surrey,
April 12, 1902.

ON MINERALS OCCURRING IN AUSTRALIAN BAT GUANO.

By R. W. EMERSON MACIVOR, F.I.C., &c.,
Formerly Assistant Professor of Chemistry, Anderson's College, Glasgow; Consulting Chemist to the Royal Agricultural Society of Victoria, Australia; &c.

SOME years ago I contributed a paper to the CHEMICAL NEWS (vol. lv., p. 215), in which I described the bat guano which forms extensive deposits in certain basaltic caves in the vicinity of Skipton, 30 miles south-west of Ballarat, and also gave an account of a number of interesting and well-defined minerals which occur in it. I propose in this communication to supplement the statements I then made by some new facts, particularly in respect to the two new magnesium-ammonium phosphates which I had named Dittmarite and Muellerite respectively, but which at the time I had not fully examined.

Struvite, $MgNH_4PO_4+6H_2O$, previously a rare mineral, occurs in great abundance in the form of large orthorhombic crystals in the moist depths of the guano, but rarely in the upper or drier portion of the deposits. It is sometimes associated with beautifully defined triclinic crystals of the more complex hydrous ammonium-magnesium phosphate, $Mg_2H_2(PO_4)_2.MgH_2(NH_4)_2(PO_4)_2+8H_2O$. Indeed, as stated in my earlier paper, the crystals of the latter are occasionally found adhering to those of the struvite in such a way as almost to suggest that they had grown out of the latter. But hannayite, the hydrous magnesium hydrogen phosphate, $MgHPO_4+3H_2O$, to which I gave the name newberyite, and the other well-defined minerals hereafter described, occur most abundantly in the seemingly older—or, at all events, drier—parts of the deposits containing the white indistinctly crystalline nodules of what appears to be altered struvite, and which approximate in composition to the normal tribasic magnesium phosphate, $Mg_3(PO_4)_2+xH_2O$. I may mention, in passing, that the aqueous extract of the guano contains, among many other salts, sodium-ammonium-hydrogen phosphate, $NaNH_4HPO_4$, which I succeeded in separating in a pure state by repeated crystallisations.

The crystallographic features of struvite, hannayite, and newberyite were years ago minutely examined and described by G. vom Rath, to whom I sent, through the late Prof. Ulrich, some representative specimens of each mineral (*Ber. Nied. Ges.*, p. 11, January 7th, 1878; and January 13th, 1879; also Dana's "A Treatise on Mineralogy," p. 832). I may take this opportunity of correcting a slight—but to me not altogether unimportant—error in connection with the discovery of hannayite into which vom Rath was led by a misstatement made in writing to him by Professor Ulrich, and which is repeated in Dana's great work of reference in a manner that reflects somewhat upon myself as a mineralogical chemist. The mineral is described as having been "discovered by MacIvor, of Melbourne, . . . and recognised as new by Ulrich, as stated in a letter to vom Rath." Now the facts of the case were that, by the time Ulrich came to my laboratory to obtain the specimens for Professor vom Rath, I had not only repeatedly analysed the mineral and by other means identified it as new, but had actually named it after my old friend and early associate Mr. J. B. Hannay. Ulrich had nothing whatever to do with its identification as a new species.

The following are some later analyses of—(A) struvite, (B) hannayite, and (C) newberyite from Skipton guano:—

	(A).		(B).		(C).	
	Found.	Theory.	Found.	Theory.	Found.	Theory.
H_2O ..	44·09	44·03	28·51	28·69	36·31	36·21
MgO* .	16·51	16·38	18·76	18·87	22·96	22·99
$(NH_4)_2O$	10·58	10·61	8·10	8·18	—	—
P_2O_5 ..	28·82	28·98	44·63	44·26	40·73	40·80
	100·00	100·00	100·00	100·00	100·00	100·00

* Including equivalent MgO for FeO and MnO present in the three minerals:—(A). FeO, 0·81; MnO, 0·16. (B). FeO, 0·310; MnO, 0·087. (C). FeO, 0·85; MnO, 0·021.

Dittmarite is met with in the form of small rhombic transparent crystals, which do not readily lose water on lengthened exposure to the atmosphere, but give up much of their water of crystallisation and lose transparency on being maintained for a time at 100—105°. On ignition, it yields magnesium pyrophosphate. Its composition indicates the somewhat extraordinary formula $MgNH_4PO_4.2Mg_2H_2(PO_4)_2+8H_2O$.

	Found.	Theory.
H_2O	23·42	23·65
MgO*	26·13	26·28
$(NH_4)_2O$	3·94	3·42
P_2O_5	46·51	46·65
	100·00	100·00

* Including MgO equivalent to 0·38 FeO and 0·08 MnO.

This interesting mineral I named in honour of my old and esteemed principal, the late Professor W. Dittmar, F.R.S.

Muellerite is yet another remarkable ammonium-magnesium phosphate found here and there sparsely distributed in the older and moderately moist (about 18 to 25 per cent H_2O) guano deposits. It occurs in small flat crystals of somewhat indistinct character, and which are with difficulty freed from adhering and locked up guano. However, after the exercise of considerable patience, I succeeded in separating some fairly clean crystals, which on drying at 100° C. for two hours did not lose water of crystallisation, but at 120° C. commence to lose weight. On strong ignition the mineral left magnesium metaphosphate, $Mg(PO_3)_2$, and, on analysis, gave numbers indicating the formula $Mg(NH_4)_2H_2(PO_4)_2+4H_2O$:—

	Found.	Theory.
H_2O	27·55	27·77
MgO*	12·42	12·35
$(NH_4)_2O$	16·15	16·05
P_2O_5	43·88	43·83
	100·00	100·09

* Including MgO equivalent to 0·20 FeO and 0·05 MnO.

This mineral I called Muellerite, in compliment to the illustrious Australian scientist, the late Baron Sir Ferdinand von Mueller, K.C.M.G., F.R.S., with whom I was much associated during my eleven years' work at the Antipodes.

The chalk-like white nodules, incidentally referred to in my earlier paper and also in this note as being "altered" struvite, and which are found abundantly distributed throughout the mass of some of the drier guano deposits, appear to consist largely of imperfectly crystalline tribasic magnesium phosphate, $Mg_3(PO_4)_2$. However, neither the chemical nor crystallographic data at present in hand will permit of my stating that this nodular material is really a distinct mineral species, but I hope soon to have an opportunity of settling the point.

In order to show the general composition of the "drier" guano deposits, in which hannayite, newberyite, dittmarite, muellerite, and the nodular mineral most usually occur, I quote the following analysis :—

Water	19·80
Organic matter*	52·83
Ash†	27·37
	100·00

The Laboratory,
29, Fenchurch Street, E.C.

METHOD FOR THE ESTIMATION OF THE TOTAL ALKALI, THE FREE ALKALI, AND THE CARBONATED ALKALI IN SOAPS.

By R. HENRIQUES and O. MEYER.

One single process can be used for determining the free and carbonated alkalis, which are usually present in small quantities only (2 to 5 per cent), at the same time as the alkalis of the fatty acids, in most household soaps. This process is based on the use of absolute alcohol, which dissolves the alkaline soaps and the free alkali, while apparently leaving the carbonated alkali intact. Unfortunately, this latter also is fairly soluble in absolute alcohol, and, further, the large percentage of water present in all soaps does not allow of the direct application of the method in question to soap. It is indispensable to thoroughly dry the soap under examination, and many other difficulties have also to be surmounted. Again, and this is the weakest point of the method, the free alkali becomes partially carbonated, which leads to results altogether erroneous.

The small apparatus, of which the following is a description, enables us to determine in a very simple manner and with great accuracy, not only the free and carbonated alkali, but also a certain number of other constants, in such a way that a complete analysis of a soap can be effected very rapidly and without difficulty. It consists of an Erlenmeyer matras of about 400 c.c. capacity, fitted with a cork pierced with three holes. One of these holes takes a small funnel, of about 100 c.c., furnished with a tap, the others take respectively the inlet and outlet air tubes. The air passing through the inlet tube is previously freed from its carbonic acid by being passed through a wash-bottle filled with strong soda-lye; the air is next passed through a drying flask filled with concentrated sulphuric acid. This apparatus is completed by a chloride of calcium tube, and a tared potash tube to retain the carbonic anhydride resulting from the decomposition of the soap. Finally, a 3 litre aspirator fitted with a syphon and pinchcock completes the apparatus.

The following is the method of working we adopted:—From 5 to 10 grms. of the soap are placed in the Erlenmeyer matras; this is dissolved in about 100 c.c. of warm water, and allowed to cool; the matras is then connected with the rest of the apparatus, and after making sure that the whole system is air-tight, dilute hydrochloric acid is run in through the funnel. Some excess of acid may be used when only the carbonated alkali is to be estimated; but if we also wish to determine the proportion of free alkali present, it is necessary to use an accurately measured volume of demi-normal acid. In all cases, however, this volume must be more than that actually needed for the complete decomposition of the soap. If the sample taken is 5 to 10 grms., 50 c.c. of demi-normal acid will suffice. The acid is run into the soapy liquor, the funnel rinsed, the tap closed, and a slow current of air allowed to pass through the apparatus. The matras is then heated gently, and it is the intensity of the heat which governs the regularity of the decomposition of the soap. The flame of the Bunsen burner should be regulated to about the size of a nut. After from thirty to forty-five minutes, provided the decomposition has proceeded regularly, the fatty acids ought to collect in a clear melted layer, and the whole of the liquid should be *almost* boiling.

As soon as the first drops of condensed steam reach the outlet tube the flame is removed, and the current of air allowed to pass for thirty or sixty more minutes, to completely drive out all the carbonic anhydride and bring it to the potash apparatus. It then suffices to weigh the latter to determine the weight of carbonate.

If, at the same time, we wish to determine the free alkali, we must continue in the following manner:—After absorbing the whole of the carbonic anhydride, the apparatus is disconnected, the funnel is well washed, the wash-water being allowed to run into the soapy liquor, which is once more brought almost to the boiling-point, and we continue the determination by Hehner's method; that is to say, the insoluble fatty acids are collected on a filter, and washed with warm water until the washings are no longer acid. The total aqueous solution is then titrated with demi-normal soda, taking into account the amount of demi-normal hydrochloric acid used for the decomposition of the soap.

In this manner we arrive at the *total alkalinity*.

The insoluble fatty acids are now dissolved in warm alcohol, and the alcoholic solution obtained is titrated

* Nitrogen (nearly all nitrates), 2·98.

† The ash contained—

Silica (and insol. in HCl 1·11 sp. gr.)	3·65	K_2O	1·79
Fe_2O_3, Al_2O_3	5·23	P_2O_5	5·38
CaO	2·00	SO_3	1·57
MgO	0·58	SiO_2 (sol.)	1·06
Na_2O	2·93	CO_2	2·00

The ash contained only a mere trace of manganese—a remarkable fact when we note the more than appreciable quantity of that substance contained in each of the minerals occurring in the guano.

with an alcoholic solution of demi-normal soda, which gives the alkali combined with the fatty acids.

Let us call *a* the total alkalinity, *b* the alkali due to the carbonic anhydride, and *c* the alkali due to the fatty acids; the whole expressed as NaHO or KHO, in grms. or parts per cent of soap, it is evident that the value *a b c* represents the free alkali; the silicates and borates being neglected in the case with which we are dealing.

The results obtained by the method described are very exact, and a complete analysis can be made in two or three hours. Further, it allows of the calculation or determination of other constants at the same time. The neutralised alcoholic solution of the fatty acids gives the proportion of *pure soap*, the most important indication in the valuation of a soap.

It is sufficient to evaporate this alcoholic solution, then to weigh the residue obtained, and to deduct from it the proportion of alkalis found, to get the percentage of fatty acids, and we should know at the same time the acid value and the mean molecular weight of these latter bodies.

We may add, however, that in some cases our process does not give results free from all suspicion. When we have to deal with soaps containing fatty acids soluble in warm water, Hehner's method is not sufficient, the total acidity may still be estimated, and that, either by means of a Huggenberg burette, or with an ordinary separating funnel.

The soap is decomposed by means of a weighed quantity of etherised acid, the acid-lye is removed, and by neutralising the etherised solution by means of a titrated alkaline-lye, the *pure soap* is obtained, which allows of the determination of the proportion of *total fatty acids.—Zeit. f. Angew. Ch.*, 1900, p. 785.

REMARKS ON COMMERCIAL PEROXIDE OF HYDROGEN.

By. G. ARTH.

It is advisable to call the attention of those who buy and use peroxide of hydrogen to some peculiarities regarding the analysis of this product.

I. It has been stated recently that some manufacturers add to their peroxide of hydrogen a certain quantity of oxalic acid. As in practice the richness of the product is always determined volumetrically by means of permanganate of potash in the presence of an excess of sulphuric acid, this addition would have the effect of decolorising a larger quantity of the indicator than that corresponding to the actual amount of H_2O_2, and thus augmenting the value in a fictitious manner. Further, this practice is unexpected *à priori*, as it is well known that oxalic acid and peroxide of hydrogen decompose each other reciprocally. Further on I shall lay stress on this point.

The method of analysis recommended to purchasers to detect this falsification is as follows:—To 100 or 200 c.c. of peroxide of hydrogen in a large foot-glass, we gradually add the equivalent volume of distilled water and pure ammonia until the reaction is distinctly alkaline (in practice *several cubic centimetres* of concentrated ammonia), then an excess of a neutral solution of chloride of calcium. If there is any oxalic acid or alkaline oxalate present in the peroxide of hydrogen, we obtain, after stirring the liquid vigorously, a granular crystalline precipitate of oxalate of lime, which eventually adheres strongly to the sides of the glass. But this precipitate of oxalate of lime may be mixed with a more or less considerable quantity of sulphate or phosphate of lime, and thus not have this characteristic appearance. In this case the precipitate is filtered and washed with distilled water containing ammonia, until the wash-waters, acidulated with sulphuric acid, no longer decompose permanganate of potash. The precipitate is then treated with very dilute warm sulphuric acid, and, in the limpid solution, oxalic acid is tested for qualitatively with permanganate which is decolorised, or even estimated with the help of the same reagent, after diluting the whole to a known volume, and taking an aliquot part.

I have had occasion to examine a large number of samples of commercial peroxide of hydrogen, and in one sample, by means of the above process, I detected the presence of a fairly large proportion of oxalic acid. A closer study of this question has shown me, however, that this method of analysis is inexact, and may lead to the apparent detection of oxalic acid where it does not exist. The following is what I have observed with regard to this matter:—

(*a*). When we wash the precipitate obtained by the ammonia and chloride of lime, we find that the washings, acidulated with pure sulphuric acid, are never entirely without action on the indicator; they always decolorise it a little—not much it is true, but still they do decolorise it, no matter how prolonged the washing has been. Thus the precipitate is dissolved or slightly decomposed on contact with ammoniacal water, a property which is not possessed by oxalate of lime. Further, if the washing is done with distilled water only, the filtrate is always slightly alkaline, even when Nessler's reagent no longer gives any reaction.

(*b*). If we now examine the very dilute sulphuric solution in which the precipitate is dissolved, we first of all notice that it decolorises the permanganate *immediately in the cold*; this does not occur with oxalic acid.

The acid solution neutralised with ammonia remains limpid for an indefinite time; if it contained oxalic acid and sulphate of lime in solution, oxalate of lime should be re formed (this is exactly what does happen if we add a trace of oxalic acid after neutralisation).

This acid solution, treated with permanganate of potassium, gives off pure oxygen free from carbonic acid, a compound which would not fail to be evolved in the presence of oxalic acid.

Finally, this sulphuric solution gives the reactions of peroxide of hydrogen very distinctly (with chromic acid and ether, &c.).

I may add that the calcic precipitate (a so-called mixture of oxalate with a little phosphate of lime), which is at first brilliant and transparent, effloresces and becomes opaque on contact with dry air; after drying at 100°, or *in vacuo*, it has a strong alkalinity which cannot be accounted for by a mixture of oxalate and phosphate of lime.

We are thus brought to the conclusion that this alkaline precipitate, which is slightly soluble in water and capable of reproducing peroxide of hydrogen under the action of dilute sulphuric acid, is nothing other than hydrated binoxide of calcium, and not oxalate of lime as supposed. The error is excusable, as we should not have expected this binoxide to be precipitated under these conditions. I further propose to return to this method for the production of binoxide of lime, which is obtained so easily with pure distilled peroxide of hydrogen. The precipitate answers to the formula $CaO_2.8H_2O$, which is already known.

I have further verified the fact that the suspected peroxide of hydrogen, which gave 8·28 volumes by the permanganate titration, gave 8·19 volumes when decomposed by binoxide of manganese and dilute sulphuric acid; the gas evolved is entirely free from carbonic acid.

II. On the occasion of this examination I was anxious to note the effect of the addition of a certain quantity of oxalic acid to ordinary commercial peroxide of hydrogen. For this purpose I made use of a very pure sample of peroxide manufactured by Messrs. Gignoux, of Lyons. At the time of the experiment this substance titrated 10·70 volumes; a portion of it was treated with crystallised oxalic acid to the extent of 20 grms. per litre, and another portion with 10 grms. per litre. These solutions were all titrated together:—

	With 20 grms. per litre.	With 10 grms. per litre.
March 13 ..	12·20 volumes	11·30 volumes
„ 21 ..	10·19 „	10·44 „
„ 28 ..	9·50 „	10·11 „
April 5 ..	9·15 „	9·92 „

It is evident that the decomposition is rapid, especially at first; after a week only it is below its original strength. The peroxide treated with 20 grms. of oxalic acid continuously gives off minute bubbles of gas, principally during the first few days; it could not be transported, therefore, in corked vessels. The use of alkaline oxalates instead of the free acid would be easily detected by determining the weight of the residue left after drying at 100°, and it is sufficiently improbable that a cheap compound could be added, in which the oxalic acid would be sufficiently imitated, and which would resist the action of the peroxide of hydrogen, and only reveal its presence in the titration with permanganate, or even after the addition of a strong dose of caustic ammonia.

III. It now remains to make a few remarks on the manner in which certain manufacturers effect the valuation of the volume of oxygen which peroxide of hydrogen can evolve. They make the titration by means of a N/50 solution of permanganate of potassium, thus containing 3·163 grms. of this salt per litre. They operate on 2 c.c. of peroxide of hydrogen, and the number of c.c. of permanganate used, divided by 3, is taken as the titration value of the product; that is to say, to indicate the number of volumes of oxygen which 1 volume of the liquid will give.

Now, this number in reality corresponds with *nothing*. Theoretically, it requires a liquor containing 5·659 grms. of permanganate per litre when operating on 1 c.c. of peroxide of hydrogen, for the volume of the reagent used to indicate, directly, the available volume of oxygen (measured at 0° and 760 m.m.). The difference between the results given by the two solutions is not negligible; in fact, the relation between this *arbitrary* and *inexact* volume of oxygen and the *real* volume is 1/0·838; a peroxide described as 10 volumes thus only gives in reality 8·38 volumes, the difference being 19·3 per cent of the real volume.

It has been impossible to find out the reason for this method of operating. In the trade it is the rule to suppress calculations whenever possible, and not to introduce useless ones, a procedure which would be necessary if we wish to use *correctly* N/50 solution; further, it is no more difficult to prepare a solution titrating 5·659 grms. per litre than one containing 3·163 grms.

We can only hope to see this irrational practice disappear, it being a source of difficulties and disputes. Users who buy peroxide of hydrogen could easily bring about this result by insisting that manufacturers should give the *real* titration value of the product they supply.—*Moniteur Scientifique*, Series 4, vol. xv., No. 715.

THE ESTIMATION OF TUNGSTEN IN ORES.

By F. BULLNHEIMER.

The analysis of impure or undressed tungsten ores is very tedious, principally on account of the presence of phosphorus and arsenic (due respectively to the apatite and arsenical pyrites which accompany the ores), which form double compounds with the tungstic acid, and make the exact precipitation of pure tungsten compounds impossible.

The treatment with aqua regia is tiresome, and fusion with cyanide and soda is unsatisfactory.

The author recommends a preliminary fusion with peroxide of sodium and a little caustic soda in a nickel crucible; the soda increases the fluidity of the mixture and enables the same crucible to be used for about twenty assays.

The mixture may contain stannate, arseniate, molybdate, phosphate, and silicate of sodium, besides the tungstate, as well as other impurities which have no action, and should therefore be eliminated.

One to 2 grms. of the ore are mixed in a nickel crucible with 4 grms. of peroxide of sodium and 3 grms. of caustic soda; this caustic soda, in one single lump, should be placed at the bottom of the crucible, which is gently heated over a Bunsen burner until the whole mass is softened; the flame is then increased until the whole mass becomes completely fluid and the bottom of the crucible commences to look red. The wolfram is quickly and completely dissolved by this method, while the tin-ore remains partly unchanged.

After solidification, the still hot crucible is placed in a vessel containing water, and the solution obtained is transferred to a flask of about 250 c.c. capacity.

If any manganate is present, the green colour can be made to disappear by means of peroxide of hydrogen.

Once cool, the flask is filled with water up to the mark, and half the solution is filtered.

Twenty grms. of nitrate of ammonium are then added (this suffices to convert the whole of the caustic soda into nitrate, giving at the same time a marked excess of free ammonia). After solution, the mixture is allowed to stand so as to precipitate the silicic and stannic acids; the necessary amount of nitrate of magnesium is then added in small quantities with continual stirring to precipitate the arsenic and phosphoric acids which may be present. The salt of magnesia should not be added until the silicic and stannic acids have been removed, or else tungsten would be found in the precipitate.

It is necessary to use nitrates of ammonia and magnesia, as the chlorides or sulphates would interfere with the subsequent use of mercurous nitrate.

After standing for from six to twelve hours the precipitate is filtered, then washed, first with ammonia, and then with water. The filtrate is made slightly acid with nitric acid, and, after cooling, if the acid has caused any appreciable rise of temperature, 20 or 30 c.c. of mercurous nitrate are added. To prepare this solution, 200 grms. of $HgNO_3$ are gently heated with 20 c.c. of nitric acid and a little water; this is then diluted up to 1 litre, and mercury added. Some hours later, ammonia is added until the solution is only faintly acid; the whole is then allowed to stand until the supernatant liquid becomes quite limpid.

The precipitate is then filtered and well washed with water containing mercurous nitrate.

If the above instructions are carefully carried out, the filtering as well as the washing can be done without any trouble.

The precipitate is then calcined, first over a Bunsen burner and then before the blowpipe until the weight is constant.

When there is much molybdenum present, which, however, is rarely the case, some time may elapse before this metal is entirely volatilised, and the weight no longer changes. The end of the operation can be hastened if, after having first heated strongly, we add a little chloride of ammonium. The heat is then applied again strongly, first with the crucible covered, and afterwards with the cover off.—*Chem. Zeit.*, xxiv., 870.

Action of Halogenated Ethers on the Sulphocarbonic Compounds of the Secondary Amines.—Marcel Delepine.—The author describes a very simple method for the preparation of the bisubstituted azoic thiosulphocarbamic ethers. He then examines the chief properties of these ethers and their relative stability. By the same method he proposes to obtain the urethanes R.NH.CSSR' and NH_2.CSSR, whose fragility and ease of reaction contrasts with the resistance of the dithiourethanes.—*Comptes Rendus*, cxxxiv., No. 12.

THE BOILING-POINT CURVE FOR MIXTURES OF ETHYL ALCOHOL AND WATER.*

By WILLIAM A. NOYES and R. R. WARFEL.

It has long been known that it is impossible to obtain absolute alcohol from dilute alcohol by distillation. Some years ago Le Bel (*Comptes Rendus*, lxxxviii., 912) showed, also, that a 98 per cent alcohol could be separated by fractional distillation into an alcohol of 97·4 per cent (by volume), and a residue containing 99·5 per cent. While these facts demonstrate that the boiling-point of absolute alcohol must be higher than that of alcohol slightly diluted, no one, so far as we can learn, has proved this by means of a direct determination.

The determination of the boiling-point curve for alcohol-water by J. K. Haywood (*Journ. Phys. Chem.*, iii., 318), has come to our notice since reading the proof of this article. Haywood entirely overlooked the minimum point. His curve, from alcohol of 85 per cent down, agrees satisfactorily with ours.

The boiling-point apparatus of H. C. Jones (*Am. Chem. Journ.*, xix., 581) was used for the determinations. The tube of the condenser was sealed to the side tube of the apparatus, and was protected from the entrance of moisture at the top by means of a tube filled with calcium chloride. A small glass tube was inserted through the cork beside the thermometer, for the introduction of water or alcohol.

The alcohol used was rendered absolute by boiling twice with quicklime, the second time with the addition of a small amount of barium oxide. In distilling it, the first and last portions were rejected. The specific gravity of the alcohol indicated a strength of 99·98 per cent.

The boiling-points for alcohol from 100 per cent to 64 per cent were determined with a Beckmann thermometer graduated to $\frac{1}{10}$°. Those from 64 per cent to 0 per cent were determined with a normal Green thermometer graduated in 0·1°. To determine the value of the degrees of the Beckmann thermometer the apparatus was connected with a large flask and a manometer, and the alcohol was boiled under diminished and also under increased pressure. In this way it was found that a difference of 1 m.m. in pressure caused, at atmospheric pressure, a change of 0·0333° in the boiling-point. Ramsay and Young's tables give 0·0339° for 1 m.m. (*Journ. Chem. Soc.*, xlvii., 640). The value of the degrees was accordingly taken as being sufficiently accurate for the present purpose.

To find the value of the Beckmann readings in terms of the true thermometer scale, pure water was placed in the apparatus, and it was connected with a large bottle and a manometer. The pressure was then reduced till the water boiled at about 80° of the true scale and a series of readings of thermometer, barometer, and manometer were taken. The barometer and manometer readings were corrected to 0° and by comparison with Regnault's tables for the vapour-pressure of water, the point on the true thermometer corresponding to the readings of the Beckmann thermometer was determined. From this value the boiling-point of absolute alcohol under a pressure of 760 m.m. is, as determined by us, 78·33°. The boiling-point calculated from the tables of Ramsay and Young (*loc. cit.*) is 78·30°. As Ramsay and Young doubtless had much more accurate means than we could command for measuring heights of manometer and barometer, we have based our tables on their value for the boiling-point, instead of our own.

The barometer used was of the syphon form. The scale was compared with a standard scale by means of a dividing engine before it was filled, and the necessary correction applied. It was filled by ourselves, the mercury being boiled under diminished pressure during the filling.

In carrying out a series of determinations a weighed amount of alcohol or of water, or, in some cases of dilute alcohol, was put into the apparatus and the boiling-point determined. A weighed amount of water or alcohol was then added, and the determination repeated. Each time, the barometer and its temperature were also read. The results were then corrected to a basis of 760 m.m. by adding or subtracting $\frac{1}{30}$° for each 1 m.m. difference in pressure. The nature of the readings will be apparent from the following illustration. In correcting the barometer readings a plus correction of 1 m.m. for error of scale is included.

SERIES 7.

Grm. of water added.	Barometer.	Temperature.	Barometer corrected to zero.	Beckmann readings.	Beckmann readings corrected to 760 m.m.	Per cent of alcohol.
0·0000	737·6	19·5	736·13	2·948	3·744	99·98
0·2128	737·9	20·0	736·37	2·925	3·713	99·51
0·2440	737·95	20·0	736·42	2·907	3·693	98·97
0·1790	738·0	18·0	736·72	2·895	3·671	98·58
—	—	—	—	—	—	—
—	—	—	—	—	—	—
0·2900	743·5	17·5	742·28	3·089	3·680	92·42
0·2860	743·7	17·5	742·48	3·115	3·699	92·10

Started with 44·478 grms. of alcohol of 99·98 per cent.

Six series of experiments, made before the use of the apparatus had been sufficiently mastered, were rejected. Four series which gave fairly concordant results were used to obtain mean values for the range from alcohol of 100 per cent to 92 per cent. From these series the following values for each change of $\frac{1}{2}$ per cent in the amount of water were calculated by interpolation :—

Per cent of alcohol.	7.	8.	10.	11.	Mean.	
100·0	3·745	3·738	3·756	3·759	3·750	
						0·030
99·5	3·714	3·708	3·727	3·730	3·720	
						0·027
99·0	3·695	3·680	3·700	3·698	3·693	
						0·021
98·5	3·669	3·655	3·687	3·678	3·672	
						0·017
98·0	3·653	3·639	3·674	3·654	3·655	
						0·016
97·5	3·638	3·625	3·658	3·634	3·639	
						0·008
97·0	3·620	3·619	3·651	3·632	3·631	
						0·004
96·5	3·624	3·612	3·643	3·629	3·627	
						0·003
96·0	3·626	3·613	3·633	3·625	3·624	
						0·002
95·5	3·627	3·613	3·634	3·632	3·626	
						0·001
95·0	3·625	3·611	3·638	3·633	3·627	
						0·009
94·5	3·636	3·623	3·647	3·639	3·636	
						0·009
94·0	3·635	3·630	3·660	3·655	3·645	
						0·016
93·5	3·653	3·644	3·678	3·668	3·661	
						0·016
93·0	3·672	3·656	3·698	3·682	3·677	
						0·014
92·5	3·679	3·667	3·713	3·705	3·691	
						0·018
92·0	3·694	3·685	3·728	3·728	3·709	

The remainder of the boiling-point curve possesses less interest, and it was not attempted to secure the same degree of accuracy. From alcohol of 92 per cent to 65 per cent two series of determinations were made and the Beckmann thermometer was used.

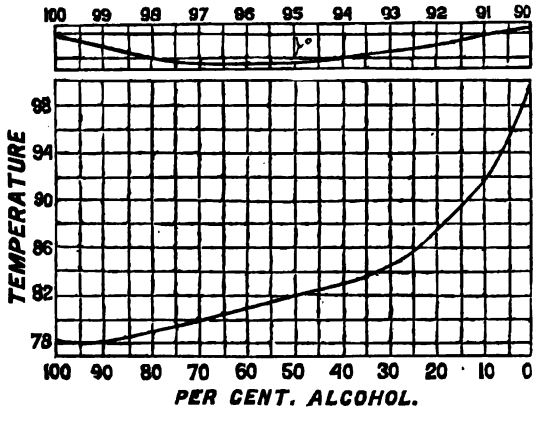
For alcohol from 65 per cent to 0 per cent, two series were made and the normal Green thermometer was used. As a part of the stem of the thermometer was outside of the apparatus, a stem correction from the tables of Rimbach was used (*Ber.*, xxii., 3075). The correction for the thermometer at 100° was determined in the apparatus with pure water, and the correction at 80° was determined by comparison with the Beckmann thermometer, whose value had been determined as described above. As the two corrections were different a table of corrections for intervening degrees was calculated on the supposition that the scale of the thermometer was uniform.

The final results of all the series are as follows:—

* An account of the work here described was presented to the Faculty of the Rose Polytechnic Institute as a thesis for the Degree of Bachelor of Science. From the *Journal of the American Chemical Society*, xxiii., No. 7.

Per cent of alcohol.	Boiling-point.	Per cent of alcohol.	Boiling-point.
100·0	78·300	78·0	79·214
99·5	78·270	77·0	79·354
99·0	78·243	76·0	79·404
98·5	78·222	75·0	79·505
98·0	78·205	73·0	79·683
97·5	78·191	71·0	78·862
97·0	78·181	69·0	80·042
96·5	78·179	67·0	80·237
96·0	78·174	65·0	80·438
95·5	78·176	63·0	80·642
95·0	78·177	55·0	81·77
94·5	78·186	48·0	82·43
94·0	78·195	37·0	83·76
93·5	78·211	35·0	83·87
93·0	78·227	29·2	84·86
92·5	78·241	26·0	85·41
92·0	78·259	22·0	86·11
91·0	78·270	20·0	87·32
90·0	78·323	18·0	87·92
89·0	78·385	13·0	90·02
88·0	78·445	10·0	91·80
87·0	78·530	8·0	93·10
86·0	78·575	7·0	93·73
85·0	78·645	5·50	94·84
84·0	78·723	4·50	95·63
83·0	78·806	3·0	97·11
82·0	78·879	2·0	98·05
81·0	78·968	1·50	98·55
80·0	79·050	1·00	98·95
79·0	79·133	0·50	99·65

In the determination with small amounts of alcohol the readings of the thermometer were taken when the vapours first entered the condenser, as after boiling for a few minutes a relatively large proportion of the alcohol present would be found in the upper layers and in the condenser, and the thermometer under those conditions registered about 0·3 higher. It was shown by slow heating that the difference was not due to the lag of the thermometer.



An examination of the table and curve shows that the minimum boiling-point is for alcohol of 96 per cent by weight. This corresponds to 97·45 per cent by volume. Le Bel (*loc. cit.*) gives the minimum as at 97 per cent (presumably by volume, though he does not make this statement). His method would tend to place the minimum too low, unless moisture was very carefully excluded.

In the accompanying figure the upper part gives the boiling-point curve for alcohol of 90 to 100 per cent on an enlarged scale. The lower portion of the figure gives the complete curve.

It will be seen that the curve is steeper on the side toward absolute alcohol than on the other side. Alcohol of 90·7 per cent has the same boiling-point as absolute alcohol. An inspection of the curve also shows very clearly why it is easy to concentrate alcohol by distillation to a strength of 30 or 40 per cent by weight, while the further concentration is relatively difficult.

A NEW INVESTIGATION CONCERNING THE ATOMIC WEIGHT OF URANIUM.*

By THEODORE WILLIAM RICHARDS
and
BENJAMIN SHORES MERIGOLD.

(Continued from p. 178).

Preliminary Work upon the Preparation, Properties, and Methods of Analysis of some Uranium Compounds.

In view of the well-known advantages of the halogen compounds for accurate analysis, when these compounds can be prepared and weighed in a state of purity—it seemed desirable to use a halogen compound as the basis of a determination of the atomic weight of uranium.

Of the four chlorides of uranium known to exist, none can be prepared in a state of purity that is beyond question. Green uranous chloride, UCl_4, which results from passing dry chlorine over a mixture of uranium oxide and carbon at red heat, is easily converted to the pentachloride, UCl_5, by further action of chlorine at high temperatures. There can be no positive evidence that the green chloride would not contain some of the pentachloride, and if the attempt is made to prepare the pentachloride from the green chloride, it is equally difficult to be sure that the conversion is complete. The trichloride, UCl_3, is made by reducing the tetrachloride with hydrogen, and here again it is difficult to be sure that the tetrachloride is completely reduced. Uranyl chloride, UO_2Cl_2, cannot be prepared in the dry state.

It is extremely probable, then, that any of the chlorides will contain larger or smaller quantities of a higher or lower chloride. It may be observed, in this connection, that Zimmermann used the chlorides in his vapour density determinations, and his analyses show good agreement. This does not show conclusively, however, that his material was free from small but fairly constant quantities of higher or lower chlorides as impurities.

On the other hand, bromine forms with uranium only three distinct compounds: the tribromide, UBr_3; uranous bromide, UBr_4; and the oxybromide, or *uranyl* compound, UO_2Br_2. The tribromide can be produced only from the tetrabromide by the action of reducing agents. Uranyl bromide, UO_2Br_2, has been certainly formed only in solution, resulting in hydrated crystals. It has never been definitely obtained in anhydrous form. Zimmermann made many attempts to form the pentabromide corresponding to the pentachloride, by passing bromine at high temperatures over sublimed uranous bromide. Every attempt gave negative results, showing that at temperatures up to the subliming point of uranous bromide higher bromides cannot exist. Since higher bromides are non-existent under the conditions prevailing in the formation of the tetrabromide, the objections to the use of the tetrachloride are not applicable in the case of uranous bromide. The investigations of Zimmermann (*Ann. der Chem.*, ccxvi., 3) have shown that the tetrabromide can be formed in an apparently definite state. It seemed probable, therefore, from the literature on the subject, that in uranous bromide we had a compound well suited to the purposes of our investigation.

The method of preparation followed at first was essentially that described by Zimmermann (*Ann. der Chem.*, ccxvi., 3). In an apparatus constructed wholly of glass, a mixture of dry nitrogen and bromine vapour was passed over a mixture of the green oxide of uranium,

* Contributions from the Chemical Laboratory of Harvard College. From the *Proceedings of the American Academy of Arts and Sciences* xxxvii., No. 14.

U_3O_8, and pure carbon. The air was first thoroughly swept out of the apparatus by a current of nitrogen, and the oxide was heated to a high temperature. When the bromine vapour was passed in, uranous bromide formed, and sublimed in brilliant crystalline plates of a brownish colour. After cooling in a current of nitrogen, the sublimate was transferred to a weighing bottle. At this point, however, unexpected difficulties arose, owing to the rapid oxidation of the bromide. Uranous bromide is extremely deliquescent, and forms with water and oxygen the oxybromide, with liberation of hydrobromic acid. Consequently, when exposed to the moist air of the laboratory, even for the short time required for removing the sublimate from the combustion tube, the bromide loses its brilliant lustre, and assumes a dull, greenish yellow appearance, due to formation of the oxy-salt. If not protected from further action of moist air, the salt liquefies completely in a surprisingly short space of time.

In an attempt to change the coating of oxybromide back to the normal salt, recourse was had to the method which has been used successfully in many atomic weight investigations carried on in this laboratory. The salt was transferred to a platinum boat, and placed, with a weighing bottle of suitable size, in a glass bottling apparatus. (For a description of this apparatus see *Proc. Amer. Acad. Arts Sci.*, xxxii., 59). A stream of dry hydrobromic acid gas was then passed over the bromide at a temperature just below the subliming point of the salt. This treatment, however, fails to restore the original brilliant appearance of the freshly sublimed bromide. The yellow colour of the oxybromide still remains. Apparently the oxybromide, once formed, cannot, by this method, be reduced to the normal uranous bromide.

In the previous investigations upon zinc, magnesium, nickel, and cobalt, in which this method of converting oxy-salts to the normal compounds has been used, the presence of even minute quantities of oxy-salt was made known by the opalescence of the solutions on account of the insolubility of these salts. With uranium, however, this method of detecting the presence of uranyl bromide cannot be used, for the oxybromide of uranium is even more soluble than uranous bromide.

The analysis of uranous bromide presents further difficulties. All uranous salts reduce silver nitrate. When a solution of silver nitrate, slightly in excess of the calculated amount, is added to a solution of uranous bromide, the silver bromide first precipitated is probably mixed with metallic silver; for if the silver bromide is filtered off, and the filtrate set aside, finely divided metallic silver soon separates. If a large excess of silver nitrate is added to the uranous bromide, a brilliant purple precipitate is obtained. It is possible that the precipitate may be a mixture of finely divided metallic silver and argentic bromide, or perhaps of normal argentic bromide and the long sought sub-bromide. Although this is an interesting phenomenon, it was not considered advisable to interrupt the research at this period for the length of time necessary for an investigation. The addition of nitric acid prevents the formation of this coloured precipitate, but owing to the danger of the loss of bromine, this is not an advisable expedient. Of course, it is possible to determine the bromine by first precipitating the uranium and adding silver nitrate to the filtrate, but this introduces a complexity of operations incompatible with the degree of accuracy requisite in an atomic weight investigation.

On account of these formidable difficulties in the preparation and analysis of pure uranous bromide, it was thought best to search for some compound which offered fewer obstacles. It will be seen that this search was vain, although it required many months.

In view of the great tendency of uranous bromide to oxidise under ordinary conditions, the use of uranyl bromide seemed to offer the simplest solution of the problem. Anhydrous uranyl bromide has never been prepared in a pure state. In the preparation of uranous bromide, if the nitrogen used contains a little oxygen, or if traces of moisture are present, there is formed, in addition to the uranous bromide, a yellow powder, very different in appearance from the brown colour of finely divided uranous bromide. This powder has been assumed by various investigators to be the oxybromide. Owing to the fact that it is always mixed with uranous bromide, an analysis has never been obtained.

There seemed to be, however, some basis for belief that under suitable conditions of temperature, moisture, and oxygen supply, it might be possible to obtain anhydrous uranyl bromide entirely free from the uranous compound. With this end in view, the green oxide, without any admixture of carbon, was heated in a stream of bromine, also in a current of hydrobromic acid. In each case there was apparently no action whatever other than a partial and gradual reduction to the black oxide. This slight reducing action is probably not due to the gases used, in the sense of being peculiar to them, for Zimmermann has shown that this reduction takes place whenever the green oxide is heated in a current of inactive gas such as nitrogen or carbon dioxide (*Ann. der Chem.*, ccxvi., 3; see also Richards, *Proc. Amer. Acad. Arts Sci.*, 1898, xxxiii., 423).

Both moist and dry gases were used. Mixtures of these gases and air were also tried, at different temperatures, The green oxide was then reduced by hydrogen to uranous oxide, UO_2, and this was then treated with various combinations of dry and moist bromine vapour, hydrobromic acid, and air, at various temperatures. Again the results were negative. Under these conditions the bromine did not combine to the slightest extent with the uranium. Since combination fails to take place, even in the presence of considerable quantities of oxygen, there is naturally some cause to doubt that the light coloured powder above mentioned is really an oxybromide. Possibly it is, after all, uranous bromide in a different state of aggregation.

The hydrated uranyl bromide is more easily obtained. The green oxide was reduced by hydrogen to uranous oxide, suspended in water, and heated with bromine on the steam bath. After driving off the excess of bromine, uranyl bromide remains in solution. The solution may be evaporated to the consistency of a thick syrup, and even under the best conditions the yield of crystals is very small. Moreover, it is almost impossible to wash the crystals free from the mother-liquor, since they are extremely soluble in water and alcohol, and ether decomposes the compound, setting free bromine. Hence uranyl bromide was abandoned.

Of the iodine compounds of uranium, the iodate alone seemed promising. This compound has been prepared and described by A. Ditte (*Annales de Chimie et de Phys.*, 1890, Series 6, xxi., 158), who assigns to it the anhydrous formula $UO_2(IO_3)_2$. The iodate was prepared by us as follows:—

To a solution of uranyl nitrate containing much nitric acid was added a solution of iodic acid, prepared by warming finely powdered iodine with nitric acid of specific gravity 1·50. Both solutions were heated to boiling before mixing. Uranyl iodate is precipitated as a yellow, finely crystalline salt, but slightly soluble in water at ordinary temperatures. At 100°, however, if some nitric acid is added, it is possible to obtain a solution containing 10 grms. of iodate to the litre. On cooling, 2·5 to 3·0 grms. of iodate crystallise out. By re-crystallising a few times, in sufficiently large vessels, it is possible to obtain a compound in a high state of purity.

The method of preparation described above is that recommended by Ditte. Although Ditte's course of procedure was carried out as exactly as possible, the compound obtained differed from that which he describes. Instead of being anhydrous, it contained one molecule of water. Inasmuch as Ditte's statement of the amount of nitric acid which he used is extremely vague, different concentrations were tried, from a solution slightly acid up to one containing 25 per cent of strong nitric acid. In every case the hydrated compound was obtained. Ditte

did not re-crystallise his compound, but our re-crystallised product was identical with that which was only once precipitated. The analysis given is the average of ten concordant analyses of material prepared from both hot and cold solutions. Both re-crystallised iodate and that precipitated only once are represented. The method of analysis is described below.

Analysis of Uranyl Iodate.

	Found.	Calculated for $UO_2(IO_3)_2H_2O$.
	Per cent.	Per cent.
Uranous oxide	42·54	42·34
Iodic acid	54·84	54·84
Water (by difference) ..	2·62	2·82
	100·00	100·00

In determining the composition of the iodate, a weighed quantity of the substance was used, and the percentage composition by weight calculated in the usual manner. For an atomic weight determination, however, any method which involves the original weight of a salt crystallised from solution as a factor in the calculation must, of course, be avoided on account of the ever present possibility of included mother-liquor. It was necessary, then, to determine directly the ratio of iodine to uranium, or to uranium oxide. To determine the uranium, advantage was taken of the behaviour of the iodate on ignition. When heated, the iodate is decomposed, water, oxygen, and iodine being given off, leaving uranium oxide. The process was carried on in an ordinary combustion tube of hard glass, a current of dry air being passed through the tube. Since Zimmermann has shown that the green oxide undergoes partial reduction at high temperature unless in an atmosphere of oxygen (*Annalen der Chemie u. Pharmacie*, 1886, ccxxxii., 287), a stream of oxygen was finally passed through the tube. The oxide was then cooled in an atmosphere of oxygen. Treated in this way, the decomposition of the iodate is not complete. Some iodine always remained in the oxide, even when the heat was maintained for three hours at a temperature just below the softening point of the combustion tube. To correct for this amount of iodine, the oxide was weighed, dissolved in dilute nitric acid, and the iodine precipitated as argentic iodide. The amount of iodine found in this way varied from 0·1 per cent to 1·0 per cent of the total iodine, according to the duration of the period of ignition.

Iodine was determined in another sample of material exactly similar to that used for the uranium. The method was, briefly, reduction of the iodate by sulphurous acid, and precipitation with silver nitrate. Stas has shown that silver iodate can be converted completely and without loss into silver iodide by the use of sulphurous acid, and the same method applies equally well to uranium iodate. ("Untersuchungen über die Gesetze der chemischen Proportionen über die Atomgewichte u. ihre gegenseitigen Verhältnisse," J. S. Stas, Aronstein's translation, p. 69). The iodate was suspended in 200 c.c. of water acidified with 20 c.c. sulphuric acid, cooled in ice to 0°, and pure sulphur dioxide was passed in until the solution smelled strongly of this reagent. The flask was then removed from the ice and shaken occasionally. From three to four hours is required before complete reduction takes place, and the last traces of iodate go into solution. When completely reduced, silver nitrate is added, and heated to 60° in order to cause the more coherent deposition of the precipitate.* Thus it was found possible to convert the insoluble iodate into soluble iodide without loss of iodine.

In this way the ratio of uranium oxide to iodine may be determined, regardless of the presence of occluded water in the iodate used, provided that the amount of water occluded be exactly the same in each of the samples. It would obviously be more satisfactory to determine both uranium and iodine in the same sample provided a sufficiently simple method could be found.

(To be continued).

* When silver iodide is precipitated in the presence of sulphurous acid, the supernatant liquid does not become clear enough to filter, even after several days, unless heated to 60°. *Vide* Stas, "Untersuchungen," p. 69.

PROCEEDINGS OF SOCIETIES.

PHYSICAL SOCIETY.

Ordinary Meeting, April 11th, 1902.

Prof. S. P. Thompson, President, in the Chair.

Dr. R. A. Lehfeldt exhibited an Electric Heater.

The apparatus consisted of a vacuum-jacketed glass tube containing water, which was boiled by passing an electric current through a platinum spiral immersed in the liquid. Tap water is preferable to distilled water because the small electrolytic action in the former case causes the boiling to proceed quietly. Different temperatures can be obtained by using other liquids.

Mr. Watson gave a list of liquids which he had found suitable for boiling electrically. He had used this method of obtaining a steady temperature in an apparatus for comparing thermometers.

Mr. Grant exhibited and described an Apparatus for Vapour-pressure Measurements.

The liquid of which the vapour-pressure is required is introduced into the vaccum of a syphon barometer. This is mounted alongside an ordinary syphon barometer, and the upper extremities of both are surrounded by a bath which can be kept at any desired temperature. The levels of the mercury in the open tubes are then adjusted until the upper mercury surfaces are at the same level. The vapour-pressure is then measured by the difference of level in the open tubes. By a simple modification it is easy to investigate the vapour-pressure of a liquid in presence of air. The two chief advantages of the method are:—(1) The simplification of the temperature correction, and (2) the wide range of temperature over which it can be employed with the use of a small bath.

Prof. Callendar referred to the advantages of the apparatus, and said that it appeared specially suitable for elementary laboratory measurements.

Mr. J. T. Morris showed an experiment illustrating the Use of Cathode Rays in Alternate Current Work.

The usual form of Braun tube was used, the rays falling upon a luminescent screen, and forming a blue spot. A solenoid conveying an alternating current was placed near the tube. The varying magnetic field caused the spot to oscillate about its mean position. To determine the maximum value of the current a switch should be arranged to rapidly replace the alternating current by a continuous one. The continuous current should then be adjusted until the maximum excursion of the spot is the same as before, and the value of the current read off from an ammeter in the circuit. For accurate work the frequency of the discharge from the induction coil exciting the tube should be adjusted until it is almost exactly in synchronism with the alternating current. The unsteadiness of the spot of light in the zero position limits the accuracy of the measurements. Mr. Morris has tried to reduce this unsteadiness by using an earthed aluminium diaphragm instead of a glass one.

Dr. Harker thought the spot of light shown might have been made brighter by the use of a larger coil, and said the vacuum required careful attention in order to get the best results.

Mr. Duddell pointed out that the movement of the spot about its zero position might be due to the action of the earth's magnetic field upon the varying current passing through the tube.

Mr. Wilson Noble suggested that the irregularity in

the position of the spot might be due to the irregular sparking of the coil, and the consequent irregular magnetic field acting upon the rays.

The Chairman said the movement was probably due to internal and electrostatic causes. He suggested the use of yttria as a luminescent material.

Mr. Morris, in reply, said the earth's field was too weak to account for the variable zero position. He had tried shielding the tube with an earthed copper-wire screen without any gain in steadiness.

Mr. Morris then showed an experiment on the Growth of Electric Currents in an Inductive Circuit.

An E.M.F. of 0·8 volt was applied to a coil wound on a ring-shaped laminated iron core. When the current had attained its steady value the E.M.F. was reversed, and the variations of the current strength shown by an ammeter. About twenty seconds were required for the current to attain its maximum value in the opposite direction. A secondary coil was also wound upon the same core, and the effect produced upon the growing current by the closing of this secondary circuit was shown. Mr. Morris has determined curves of growth for different currents, and he explained how similar curves could be used to determine experimentally the hysteresis loss in transformers.

Dr. Glazebrook drew attention to the fact that this method has been applied with some slight modifications to the determination of the hysteresis loss in some 3000 H.P. transformers.

Mr. Croft showed some Apparatus and Devices Useful in Teaching.

The method of determining graphically the focal length of a lens from the distances of conjugate foci from the centre was illustrated. The graphic solution of a quadratic equation was also shown.

An apparatus for producing and demonstrating the properties of three phase currents was exhibited and described.

Mr. Croft then showed crystals illustrating the five regular solids, and an electric lamp with the filament in one plane useful for optical work.

The flatness of a piece of plate-glass can be tested with a scribing block. The point is adjusted to touch the glass in one position. By breathing on the glass and moving the block about it is easily seen if the point leaves the surface.

Mr. J. M. Barr said that the method could not be applied to steel. To test the flatness of a piece of steel he had made use of a small table with four legs, placed upon the surface. One leg of the table was adjustable, and was made part of a circuit containing a microphone. If the ends of the four legs are placed in one plane it is easy to test flatness by observing when the adjustable leg leaves the surface. The amount of the irregularity of the surface can be measured by having a micrometer attached to the movable leg.

The Society then adjourned until April 25th.

OBITUARY.

ALFRED CORNU.

It is with regret that we record the death of Professor Alfred Cornu, which occurred last week in Paris.

Prof. Cornu was born on March 6, 1841, and is therefore sixty-one years of age. After completing his education at the Ecole des Mines, he, in 1867, was appointed Professor of Experimental Physics at the Ecole Polytechnic, which position he still held at the time of his death. His work was almost entirely directed to optics, including diffraction, polarisation of light, and spectroscopy, &c., though he has also published papers on acoustics, magnetism, and electrical questions.

Prof. Cornu's first paper was published in the year 1861, and his principal work, viz., on the velocity of light, was done in 1871. The amount of work he got through may be judged from the number of papers he wrote. Up to the year 1883 he wrote over seventy papers, according to the Royal Society Catalogue, besides many important ones since that date.

In 1878 Prof. Cornu was made a Member of the Academy of Sciences, and in the same year he received the Rumford Medal of the Royal Society, of which he was made a Foreign Member six years later. He was well known in English scientific circles, and has delivered three Friday Evening Discourses at the Royal Institution. The first occasion was on May 7, 1875, on "New Determinations of the Velocity of Light"; the other two lectures were delivered on May 16, 1879, and June 7, 1895; his subject on the latter occasion being "Physical Phenomena of the Upper Regions of the Atmosphere."

Prof. Cornu was a fluent and polished speaker, and a very successful manipulator and experimenter. His last lecture in England was in 1899, at the Jubilee Celebration of Sir George Stokes, his subject being "The Wave Theory of Light, and its Influence on Modern Physics."

NOTICES OF BOOKS.

Pharmacopœdia; a Commentary on the British Pharmacopœia, 1898. By Edmund White, B.Sc. (Lond.), F.I.C., Pharmaceutist to St. Thomas's Hospital, London, and John Humphrey. With Forty-six Full-page Plates. London: Henry Kimpton. 1901. Pp. 692.

The word Pharmacopedia has been adopted as the title of this volume, as it exactly expresses the intention of the authors, viz., to give "information about drugs." The scope of the work is, however, restricted to the official materia medica, otherwise it would have been too unwieldy in size. In most text-books on pharmacy the subject-matter has been composed in too technical a manner; in this volume the authors have endeavoured to make the technical details of the official monographs more easy of comprehension by reference to the elementary principles laid down in those text-books, and it is believed that students will thus be enabled to secure a better grasp of the subject which pharmacists and medical practitioners are called upon to study.

The plan of the book is simplicity itself; the drugs are taken in alphabetical order beginning with "Acaciæ Gummi," or gum arabic, and going all through the list to "Zingiber," or ginger. The drug is first described, and if a chemical product, its method of production is given; this is followed by an account of its characters, and tests for its detection and recognition, and concludes with a few general notes on its properties and peculiarities.

On page 535 we come to "Notes on the Indian and Colonial Addendum (1900) to the British Pharmacopœia, 1898." Most of the drugs have been included with the object of serving—in the districts where they are produced—as equivalents of other drugs already official in the pharmacopœia; it is not, however, intended that they should be substituted for the official drugs, except where authority is expressly given in the text. Moreover, such authority is only given in those divisions of the Empire which are specially indicated in the addendum.

After the usual metrical and other tables of reference, the volume closes with the "Pharmacopedic Atlas," comprising forty-six plates giving a large number of illustrations of crude drugs and microscopical sections. The index is very full and comprises fifty-seven columns.

Methods for the Analysis of Ores, Pig-iron, and Steel, in Use at the Laboratories of Iron and Steel Works in the Region about Pittsburg, Pa. Contributed by the Chemists in charge, and Edited by F. C. Phillips. Second Edition. Easton, Pa.: The Chemical Publishing Company. 1901. Pp. 170.

A knowledge of the exact chemical composition of all raw materials and products of manufacture is becoming of more and more importance in modern iron and steel works, as in many other industries.

Not only is accuracy required, but the cry is always for greater speed in getting the results. Steel works chemists are continuously striving to shorten the time necessary for the course of the analyses, without sacrificing accuracy of results, and it may be safely said that with the great saving of time effected by recent changes in methods of analysis, greater accuracy has been secured than was possible by the older and slower methods.

The methods described in this edition are more rapid than those of the earlier edition, and are even more accurate. They comprise those in use at twenty different iron and steel works, many of which are of world-wide fame, such as the Carnegie Steel Company, the Shenango Valley Steel Company, the Pittsburg Steel Foundry Company, and many others equally well known.

In the appendix, which covers twenty-five pages, we find about a dozen methods for special determinations, such as phosphorus in coke and coal, blast-furnace cinders and their analysis, the complete analysis of chrome ore, &c.

The whole forms a volume of great value to iron and steel works chemists, who will find considerable help from the experience of others engaged in the same class of work. The book is well indexed.

Outlines of Electrochemistry. By H. C. Jones. New York: The Electrical Review Publishing Co. 1901.

This little book consists of papers on the most important chapters of electrochemistry, reprinted from the *Electrical Review*. Starting from the basis of the relations between osmotic pressure and gas pressure, the chief facts and theories of electrochemistry are put forward in simple graphic language, specially likely to be appreciated by those whose scientific knowledge is not extensive. This is not to say, however, that the papers are in any degree "popular." The author has successfully kept before him the requirements both of the student of chemistry and the electrical engineer, and shows himself particularly able in his grasp of the difficulties likely to occur to those who are reading the subject for the first time, forestalling such difficulties by short accurate explanations; and he is an adept also in stating the gist of a question in a few concise words, so that, while a wide range of the subject is covered in short space, the book by no means presents the aspect of a mere dry emuneration of facts, but is really interesting reading.

A Travers la Matière et l'Energie. By Docteur F. E. Blaise. Paris: Librairie Ch. Delagrave.

From the preface to this astonishing book we conclude that it represents an attempt to apply the scientific argument as the basis of philosophical research, and the result appears to lead us to the conclusion that Epicurus and the alchemists were after all in the right, and that our wanderings from the paths marked out by them have only led us to hopeless error. We have before us the old hypothesis that the atoms of every element have resulted from the coalescence of particles of "primether," which is at once the basis common to both matter and energy; these particles in their coalescence obey the laws of electricity and mechanics. All properties of matter are proved to be reducible to movement.

After a good deal of discussion of the laws of electricity and of induction, we pass to the application of the laws of mechanics, &c., to moral and social problems, to a parallel between religious dogma and notions of energy and matter, and, incidentally, to a refutation of Darwinism, which was foretold in an earlier part of the work in the sentence:—"C'est l'appareil qui crée la fonction." It would be an easy, if profitless, task to point out many similar instances of unguarded or erroneous assertions; we cannot refrain, however, from calling attention to the paragraph on page 320, which embodies the author's ideas as to the descents of mules and hybrids.

We are told that the doctrine of the progressive variation of individuals derived from a common type has still, and will always have, its adherents, whose enthusiasm is the result of some species of moral blindness and obliquity. This is perhaps the mildest description applied to those who presume to differ from M. Blaise. Those who are sufficiently curious to glance through the book to extract some passing amusement from its mixture of doubtful philosophy and still more doubtful science will probably feel that at any rate they err in good company when they do not agree with much that M. Blaise tell us.

Introduction à l'Etude des Métaux. By Alfred Ditte. Paris: Société d'Editions Scientifiques. 1902.

The subject of the metallic elements is attacked in this book in a somewhat original way, likely to lead to wider and more comprehensive views than the more usual method, without, however, sacrificing a detailed knowledge. Instead of studying each metal and its compounds separately, the student first reads chapters on the geological occurrence of the metals, their origin, and extraction from their ores. Hence he passes to their general physical properties, and the causes which modify these properties. The metals are then considered in their relations to other bodies; firstly, the other elements, then acids, bases, and salts. A similar method of treatment undoubtedly simplifies the study of the carbon compounds, where a general knowledge is to be desired rather than any special attention to details, and no doubt this method will commend itself to students and teachers of inorganic chemistry. The book is strictly methodical and precise, especially as regards the general review of the subject of the action of the metals, the explanations by reference to thermochemical phenomena being excellent. The section on the persistence or disappearance of the properties of an element in various compounds would probably fail to give the student very definite views, but at any rate he would have the minimum amount to unlearn on proceeding to more exhaustive treatises or original publications.

Manuale del Chimico e dell' Industriale. By Dottor Luigi Gabba. Milan: Ulrico Hoepli. 1902.

The reception of this handbook of chemistry and allied industries has warranted the production of a third revised and enlarged edition, in which special attention is paid to analytical processes. The analytical tables of Will are included, in the hope of making the book useful to students; these are printed on twelve separate sheets, and ingeniously fastened within the cover of the book, so as to be readily detached, a great convenience for use in the laboratory. The first three chapters consist chiefly of tables of physical and chemical data, including copious lists of the densities and concentrations of salts and organic substances, and tables of solubility of various bodies in water and other solvents. Unfortunately this part of the book is by no means free from inaccuracies and misprints; we notice, for instance, that though the table of atomic weights from Lunge's "Taschenbuch" includes praseodymium and neodymium, in other places in the book didymium is still to be found amongst the elements, and other errors might be cited. The fourth chapter contains short descriptions of analytical processes relating to water, glass, oils and fats, manures, &c., and also assaying processes. This more descriptive portion con-

tains a really wonderful amount of information in a short space, and it would be unreasonable to expect fuller accounts of the various processes within the limits of the book. Much useful matter is to be found in the manual, which deserves to be largely employed as a book of reference by analytical and technical chemists and students of chemistry.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxiv., No. 12, March 24, 1902.

Direct Hydrogenation of Oxides of Carbon in presence of certain Finely Divided Metals.—Paul Sabatier and J. B. Senderens.—Cobalt when reduced from its oxide by means of hydrogen below 450°, behaves in exactly the same manner as reduced nickel both with regard to carbonic oxide and carbonic anhydride, these being regularly hydrogenated and transformed into methane. These changes do not start until a high temperature is attained. The reaction, beginning at about 300°, becomes more rapid at 360°, and still more rapid at 400°. The reaction takes place according to the equation $CO_2+H_8=CH_4+2H_2O$. Finely divided platinum effects the direct hydrogenation of carbon dioxide or carbon monoxide. When the action takes place below 420°, there is no appreciable formation of methane. The same occurs with palladium and also with iron reduced from its oxide by prolonged action of hydrogen at 500° C. Finely divided copper has no such action.

Boiling-point of Selenium and some other Pyrometric Constants.—Daniel Berthelot.—The author determines with great accuracy the boiling-point of selenium and other pyrometric constants. His results are expressed in the following table:—

Boiling-point of selenium ..	$690° + \frac{H-760 \text{ m.m.}}{10 \text{ m.m.}}$
Boiling-point of cadmium ..	$778° + \frac{H-760 \text{ m.m.}}{9 \text{ m.m.}}$
Boiling-point of zinc	$918° + \frac{H-760 \text{ m.m.}}{8 \text{ m.m.}}$
Melting-point of silver	962°
Melting-point of gold	1064°

Thermic Equivalent of Dissociation, of Vapourisation, and Heat of Solidification of Ammonia.—M. de Forcrand.—The author determines the heat of solidification of a gaseous molecule from a knowledge of the dissociation and the heat of formation of a certain number of its dissociable compounds. Such a method is of great use when direct measurement is impossible or difficult, as is usually the case with gases.

An Acid Monosodic Orthophosphate.—H. Giran.—The author examines the crystals which are recovered from the cylinder in which commercial metaphosphoric acid is prepared. He estimates directly the phosphoric acid, soda, and water, and thus deduces the formula $P_2O_5.NaH_5$. This is the monosodic salt corresponding to M. Joulie's sesquisodic phosphate.

Sesquisodic Phosphate.— J. B. Senderens.— The author further discusses M. Joulie's paper on the new phosphate of soda.

Some New Compounds of Methylene.— Marcel Descudé.— The author has already shown that zinc chloride provokes the very rapid action of trioxymethylene with an acid chloride, R—COCl, giving rise to the compound R—COO—CH_2Cl. Besides this, during the distillation of the crude product in vacuo, the product (R—COO)$_2CH_2$ is present in varying proportions, but always in considerable quantity. By repeating this reaction on a great number of the acid chlorides the author now establishes the fact that such a reaction is absolutely general. However, in the case of the bibasic acid chlorides (those of succinyl and phthalyl), when the action is very energetic, the distillation, even in vacuo, provokes complete decomposition, and, as a residue, the theoretical quantity of the corresponding anhydride is found proportionate to the weight of acid chloride employed. Finally he obtains a series of mixed compounds in accordance with the reaction—

$$R—COOCH_2Cl+R'—COOK = KCl+\frac{R—COO}{R'—COO}>CH_2,$$

which regularly occurs when the mixture is heated for at least three hours to a temperature varying with the radicle but always above 160°.

No. 13, March 31, 1902.

This number contains no chemical matter.

MISCELLANEOUS.

A New Oxide of Molybdenum, the Semipentoxide of Molybdenum.—P. Klason.—This paper deals with the examination of the various compounds in which molybdenum is pentatomic, compounds already partly announced by Berzelius and De Bray. The author proposes the name *molybdenyl* for the trivalent radical MoO, and *molybdone* for the divalent radical MoO_2. *Chloride of Ammonium and Molybdenyl*, $MoOCl_3,2NH_4Cl$.—Two hundred grms. of molybdate of ammonium are dissolved in 600 c.c. of fuming hydrochloric acid, and for each atom of molybdenum present we add 1 molecule of iodide of ammonium (or concentrated HI) and 0·1 (or 1) molecule of chloride of ammonium; this is distilled on the sand-bath, iodine is given off, and when the fumes of this latter have almost ceased to appear, the cooled residue is saturated with hydrochloric acid gas, the contents become dirty brown or green in colour. After a few hours, beautiful grass-green octahedric crystals are deposited which are drained after washing with concentrated hydrochloric acid. This body is soluble in water, with a yellowish-brown colour, but with partial hydrolysis. The solution has become partially oxidisable in the air with the formation of the molybdenum blue; the addition of hydrochloric acid brings back the green colour. Alcohol acts like water, and separates out chloride of ammonium. *Hydrate of Molybdenyl*, $MoO(OH)_3$.—The solution of the preceding salt treated with an excess of ammonia gives a precipitate, and the liquid contains a good deal of MoO_3 (De Bray). But if the operation is carried out in the cold, in dilute solution, and by gradually adding, while stirring, the strictly necessary quantity of ammonia, the molybdenum is deposited integrally in the form of a precipitate resembling ferric hydrate, but slightly lighter in colour; this is hydrate of molybdenyl, and is rather difficult to collect and drain on the filter; it is insoluble, or only slightly soluble, in alkaline solutions, but an excess of these latter alters the product, giving soluble molybdates. It has no acid properties. *Semipentoxide of Molybdenum*, Mo_2O_5.—The preceding hydrate, carefully dried in a current of carbonic acid, gives the corresponding oxide, a black-violet powder difficultly soluble in hydrochloric or sulphuric acids. *Chloride of Molybdenyl*, $MoCl_3$.—MM. Blomstrand (*Journ. f. Prakt. Ch.*, vol. lxxi., p. 466) and Puttbach (*Lieb. Ann.*, vol. cci., p. 123) obtained, by the action of the chloride on a mixture of MoO_2 and carbon, an oxychloride which should be $MoOCl_4$. The author thinks that this product consists principally of $MoOCl_3$, as, when re-dissolved in concentrated hydrochloric acid and treated with sal-ammoniac, it gives directly the double salt described at the commencement of this paper.—*Berichte*, vol. xxxiv., p. 148.

MEETINGS FOR THE WEEK.

MONDAY, 21st.—Society of Arts, 8. (Cantor Lectures). "Glass for Optical Instruments," by Richard T. Glazebrook, M.A., D.Sc., F.R.S.

TUESDAY, 22nd.—Royal Institution, 3. "Recent Methods and Results in Biological Inquiry," by Allen Macfadyen, M.D., B.Sc, Fullerian Professor of Physiology, R.I.

WEDNESDAY, 23rd.—Society of Arts, 8. "Opto-technics," by Prof. Silvanus P. Thompson, D.Sc., F.R.S.

THURSDAY, 24th.—Royal Institution, 3. "The Oxygen Group of Elements," by Prof. Dewar, M.A., LL.D., D.Sc., F.R.S., Fullerian Professor of Chemistry, R.I.

FRIDAY, 25th.—Royal Institution, 9. "X Rays and Localisation," by James Mackenzie Davidson, M.B., C.M.

—— Physical, 5. An Exhibition of a Mechanical Break for Induction Coils, by Dr. Dowson Turner. "A Temperature Indicator for Use with Platinum Thermometers, in which Readings are Automatically Reduced to the Gas Scale," by R. S. Whipple. "Note on the Compound Pendulum," by S. A. F. White.

SATURDAY, 26th.—Royal Institution, 3. "British National Song" (with Musical Illustrations), by William H. Cummings, Mus.D. (Dub.), F.S.A., Hon.R.A.M., Principal of the Guildhall School of Music.

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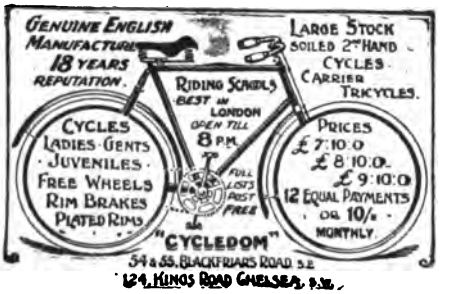

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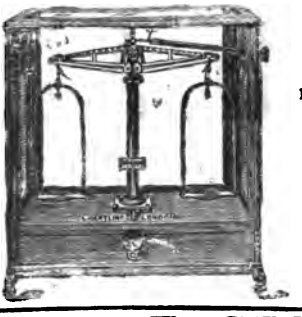

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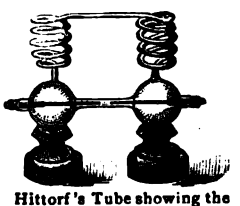

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PERSULPHURIC ACID.*

By HENRY E. ARMSTRONG, V.P.R.S., and T. MARTIN LOWRY, D.Sc.

Although it was observed by Faraday in 1834 that "if the acid were very strong, a remarkable disappearance of oxygen took place" on electrolysing aqueous solutions of sulphuric acid ("Researches in Electricity," Series vii., § 728), it was not until 1878 that its disappearance was at all satisfactorily accounted for by the discovery of *persulphuric acid* by Berthelot.

Only the anhydride, S_2O_7, was isolated by Berthelot, but he concluded that the corresponding acid was formed (1) on dissolving the anhydride in water, (2) on electrolysing strong solutions of sulphuric acid, and (3) by the interaction of hydrogen peroxide and ordinary sulphuric acid. The correctness of these conclusions appears to have been regarded as beyond question after Marshall had discovered in 1891 that well-defined salts of "Berthelot's acid" could be prepared by electrolysing solutions of potassium- or ammonium-hydrogen sulphate. Doubt arose, however, when Caro, in 1898, discovered that if Marshall's salts were acted on by sulphuric acid, a new acid was obtained having properties markedly different from those associated with Berthelot's acid. "Caro's acid" soon acquired importance as an oxidising agent, owing to the good use that was made of it by Bamberger and by von Baeyer and Villiger.

Having found that Caro's acid liberated iodine very rapidly, whereas Berthelot's acid acted but slowly on iodides, these latter chemists were able to devise a process by which the one acid could be estimated in presence of the other. By removing sulphuric acid by means of barium phosphate, they obtained a solution containing Caro's acid and Berthelot's acid, in which they determined the amount of each of these substances as well as the amount of sulphate to which the persulphuric acids gave rise when decomposed. Their results led them to conclude that the ratio of sulphur to active oxygen in Caro's acid was—

$$SO_3 : O = 1 : 1.$$

Placing the simplest possible interpretation upon this result, they assigned to the acid the formula—

$H_2SO_5 = H_2O_2 \cdot SO_3$ (*Ber. Deut. Chem. Ges.*, 1901, 853).

Meanwhile the problem had been studied from a somewhat different point of view by Lowry and West (*Chem. Soc. Trans.*, 1900, 950), who had determined the equilibrium subsisting between hydrogen peroxide and "persulphuric acid" in presence of sulphuric acid and water, and had found that the ratio which the hydrogen peroxide bore to total "persulphuric acid" was entirely dependent on the ratio which the water bore to the sulphuric acid—the ratio of hydrogen peroxide to "persulphuric acid" being ultimately the same in a mixture prepared from hydrogen peroxide and sulphuric acid as in a solution of equal strength prepared by electrolysis. The experimental curve approximated very closely to a curve deduced from an equation of the fourth order, and assuming that the chief product of interaction was a persulphuric acid of the series $H_2O_2 \cdot nSO_3$, it was to be supposed that it was the fourth term of the series, viz., $H_2O_2 \cdot 4SO_3$. But there were indications that some simpler acid was also present in small quantity.

These results applied mainly to concentrated solutions, 85 per cent of the change taking place between the limits expressed by the formula $H_2SO_4 \cdot H_2O$ and $H_2SO_4 \cdot 4H_2O$; and virtually no peroxidation of the sulphuric acid took place below the limit expressed by the formula $H_2SO_4 \cdot 6H_2O$. Von Baeyer and Villiger, on the other hand, had dealt with a product existing in dilute solution. The different conclusions deduced from the two sets of results were, therefore, not necessarily discordant; it was possible that the product examined by Baeyer and Villiger had been formed by hydrolysis from the "higher" acid which Lowry and West's observations had indicated was present in concentrated solutions. It is also to be noted that the determination of the ratio of active oxygen to sulphur is sufficient to determine the composition of the acid only in the case of the acid being one of the $H_2O_2 \cdot nSO_3$ series; obviously, other types of "persulphuric acid" are possible.

If we consider what must be the properties of the persulphuric acids generally, it is clear that whereas the salt of a dibasic acid of the formula H_2SO_5 would remain neutral on withdrawal of the peroxide oxygen, salts of higher acids would yield more or less sulphuric acid when decomposed. As a carefully neutralised solution of Caro's acid becomes acid when heated, the salt originally present in it cannot be one derived directly from the acid H_2SO_5—assuming that this is dibasic.

We are much indebted to Mr. A. J. Cook for having made a long series of determinations which show that the ratio of the increase in acidity to active oxygen lost is—

$$SO_3 : O = 1 : 2,$$

a result which finds expression in the formula $H_2S_2O_9$, but is in direct opposition to the formula H_2SO_5; thus, supposing the calcium salt to be used:—

$$2CaSO_5 = 2CaSO_4 + O_2$$

$$CaS_2O_9 + H_2O = CaSO_4 + H_2SO_4 + O_2.$$

The probability that the acids in question have the composition suggested is considerable, if the manner in which sulphuric acid may be expected to undergo electrolysis be taken into account. Strong solutions of the acid may be supposed to contain both sulphuric and disulphuric acids, and it may be expected that both would "peroxidise"; on electrolysis, the former would give perdi- and the latter pertetra-sulphuric acid, thus—

$HO \cdot SO_2 \cdot OH$	$HO \cdot SO_2 \cdot O$ $HO \cdot SO_2 \cdot \dot{O}$
Sulphuric acid.	Perdisulphuric acid.
$HO \cdot SO_2 \cdot O \cdot SO_2 \cdot OH$	$HO \cdot SO_2 \cdot O \cdot SO_2 \cdot O$ $HO \cdot SO_2 \cdot O \cdot SO_2 \cdot \dot{O}$
Disulphuric acid.	Pertetrasulphuric acid.

An acid of the formula $H_2S_2O_9$ may be regarded as the anhydro-acid derived from permonosulphuric acid, *i.e.*, as $\left.\begin{matrix} HO \cdot O \cdot SO_2 \\ HO \cdot O \cdot SO_2 \end{matrix}\right\} O$. It is scarcely necessary to say that the interpretation now put upon the data afforded by solutions of Caro's acid will need to be verified, and cannot be accepted as final until salts of the acid have been isolated. This we are engaged in doing.

Further consideration, in the light of the facts brought forward by Baeyer and Villiger and in the present communication, of the results arrived at by Lowry and West, has served only to confirm the conclusion that the chief product of oxidation of sulphuric acid by hydrogen peroxide in presence of less than 50 per cent of water is an acid richer in sulphur trioxide than perdisulphuric acid, and to justify the assumption on which they based the formula $H_2S_4O_{14}$—namely, that the acid is a member of the series $H_2O_2 \cdot nSO_3$. Lowry and West determined only the ratio of peroxide oxygen to persulphuric oxygen, and paid no attention to the total oxidising power of the solution. That they were justified in this course is apparent from the fact that no change in the ratio of the two forms of oxygen was observed on varying the strength of the

* A Paper read before the Royal Society, March 20, 1902.

peroxide solution from 10 to 40 "volumes," nor was any change observed as the oxidising power gradually decreased when the acid decomposed.

If the main product of the interaction had been an acid of the formula $H_2S_2O_9$, this would not have been the case, as the equilibrium represented by the equation—

$$\underset{C_2}{2H_2O_2} + \underset{C_3}{2H_2SO_4} = \underset{C_1}{H_2S_2O_9} + \underset{C_4}{3H_2O}$$

would depend on the total oxidising power of the solution as well as on the proportion of sulphuric acid and water; this is seen most clearly when the equation of mass action,—

$$KC_2^2C_3^2 = C_1C_4^3,$$

is transposed into the form—

$$\frac{C_1}{C_2} = K\left(\frac{C_3}{C_4}\right)^2 \cdot \frac{C_2}{C_4},$$

which indicates that the ratio C_1/C_2 of persulphuric oxygen to peroxide oxygen depends not only on the ratio C_3/C_4 of sulphuric acid to water and on the concentration C_4 of the water in the solution, but also on the actual concentration C_2 of the hydrogen peroxide. The fact that the equilibrium is independent of total oxidising power can only be explained if the chief products of interaction are members of the series $H_2O_2 \cdot nSO_3$, or hydrates thereof.

As there was no sufficient evidence to justify the assumption that a third persulphuric acid was present in the solutions they examined, Lowry and West had no alternative but to regard Caro's acid as pertetrasulphuric acid, and their simpler member of the series as the acid corresponding to Marshall's salts. The determination of the ratio of sulphur to active oxygen by Baeyer and Villiger has rendered such a limitation impracticable, and we now feel not only that it is justifiable, but that we are compelled to postulate the existence of *at least* three persulphuric acids, viz.,—

Pertetrasulphuric acid	$SO_3 : O = 4 : 1$
Perdisulphuric acid..	$SO_3 : O = 2 : 1$
Peranhydrosulphuric acid (Caro's acid).	$SO_3 : O = 1 : 1$

In carrying out his experiments, Mr. Cook nearly neutralised solutions of Caro's acid by means of a carbonate, and then neutralised the liquid by means of either sulphuric acid or caustic soda, portions being taken out and tested with methyl-orange. Measured portions of the neutral solution were run as quickly as possible into flasks containing a little dilute sulphuric acid to arrest decomposition; the persulphuric acids were then estimated by Baeyer and Villiger's method. Portions of the same solution were heated at 100° until all oxidising power was lost, and the acidity developed was estimated by caustic soda, using methyl-orange as indicator.

Solution I.—This was prepared by digesting potassium persulphate at 60—70° with a solution containing only 10 per cent of sulphuric acid. At the end of about two hours the cooled solution was diluted and neutralised with chalk. The amount of iodine liberated at once by the Caro's acid present, expressed in terms of decinormal thiosulphate solution, was equivalent to 41·54 c.c.; the amount of iodine liberated slowly by the perdisulphuric acid present was equivalent to 1·05 c.c. The acid liberated on warming the solution was equivalent to 22 06 c.c. The calculated amount, assuming the ratio $2O : SO_3$, would be $1·05 + \frac{3}{4}41·54 = 21\ 80$ c.c.

In the subsequent experiments 15 grms. of potassium persulphate was digested with from 35 to 25 c.c. of concentrated sulphuric acid at the ordinary temperature during one to two hours. The solution was then diluted with ice and neutralised: in Experiments 2, 3, 4, and 6 with chalk, in 5 with sodium bicarbonate, and in 7 with sodium carbonate, using phenolphthalein as indicator. In Experiment 7 the solution was mixed with excess of caustic soda before warming it to decompose the per-salts, and the excess was subsequently determined. No hydrogen peroxide was present except in solution 5, in which case the amount was determined by permanganate and allowed for.

The following are the results obtained in terms of decinormal solutions:—

	Caro's acid.	Perdisulphuric acid.	Acid liberated. Found.	Acid liberated. Calculated.
I.	41·54	1 05	22·1	21·8
II.	38·8	5·91	26 6	25 3
III.	35·0	2 77	20·2	20·3
IV.	39·24	2·24	21·3	21·8
V.	37·3	1·56	21·5	20·2
VI.	49·8	3·43	28·7	28·3
VII.	23·3	0·71	12·2	12·4

ON THE HEATLESS CONDITION OF MATTER:

Being an Extension of the Kinetic Theory.

By JAMES HANCOCK BRINKWORTH
and
GEOFFREY MARTIN, B.Sc.(Lond).

According to the kinetic theory, all bodies are composed of molecules in a state of very rapid motion. In a solid the molecules are not in absolute contact, but vibrate rapidly about fixed centres.

Consider a solid placed in an infinitely strong and unyielding cylinder, made out of a good heat-conducting substance, and let the whole be kept in a liquid bath at a constant temperature.

The solid is now slowly compressed. As the compression proceeds and the molecules are driven closer together, *heat* will be evolved; but the surrounding liquid will conduct this away, and if the compression be slow enough, the temperature of the whole system will remain constant. But this compression cannot continue beyond a certain amount, for ultimately the molecules will be driven into absolute contact, and will be so tightly pressed, the one against the other, as to be absolutely unable to move relatively to each other.

Therefore, in this condition the *molecules* can possess no kinetic energy or relative motion of any description.

Now, according to the kinetic theory, the temperature of a body is measured by the kinetic energy of its molecules.

So that our solid, when in the above state, has been by pressure reduced to the absolute zero of temperature. Here, then, we are dealing with a very remarkable case.

We have here a solid at the absolute zero of temperature, in the midst of a medium at a much higher temperature, and yet the solid (on account of the enormous pressure to which it is subjected) is unable to take up the surrounding temperature, but persists in its heatless state.

But if the pressure be suddenly released, then the solid immediately regains the power of acquiring heat from neighbouring bodies at higher temperatures, and its temperature will rapidly rise from absolute zero, until it reaches that of the surrounding material—following in this the law of thermal equilibrium.

It therefore appears to be a necessary consequence of the molecular theory that at a *very great pressure* matter *must* be reduced to a peculiar condition in which it will not obey the law of thermal equilibrium; and this law states that:—*If a body* A *be in intimate contact with a body* B, *and both be at different temperatures, then the bodies are in a state of unstable thermal equilibrium, and the temperature of the one will rise and the other fall until a common temperature be obtained.*

Now the pressure at which this remarkable physical condition becomes manifest depends upon the nature of the material, and will alter with the temperature. As the temperature falls, and the kinetic energy of the molecules becomes less, a smaller pressure will suffice to bring the molecules into contact. So that, in general, to each tem-

perature there must correspond a minimum pressure which just suffices to bring matter into this new condition, and a curve could be traced, did we possess the experimental data, showing the variation of this minimum pressure with the temperature, in much the same way that boiling-point and freezing-point curves can be traced.

At ordinary temperature, of course, the pressures will be terrific—probably beyond our experimental power—but at very low temperatures the case is different, and a moderate pressure will suffice to bring about this new condition. But the "pressure" here is made up of two parts—the *external* and the *internal*, the latter being due to mutual molecular attraction.

So that even if the external pressure is zero, yet *the molecules will be under a very considerable internal pressure.* And the very interesting conclusion must be drawn that to this internal pressure *alone* there must correspond a definite temperature above absolute zero, at which this new condition is induced in matter.

So that if we cool down a body under zero external pressure to the low temperature corresponding to the *internal pressure*, at this point its temperature will abruptly drop to the absolute zero.

The usual assumption, therefore, that all substances can be cooled continuously and uniformly down to the absolute zero is incorrect.

Each substance can only be cooled continuously and uniformly down to a minimum temperature, depending upon the total pressure, and any further cooling below this causes its temperature to drop more or less abruptly and of its own accord to the absolute zero, no matter whether the surrounding temperature be above absolute zero or not.

The heat energy which thus disappears going to increase *intramolecular motion.* In fact, in order to maintain a "temperature state" in matter there is required a certain minimum amount of energy, in exactly the same way that a certain minimum amount of energy is required in order to maintain matter in the "liquid" and "gaseous" state.

This minimum amount of molecular energy (contained in unit mass)—which, of course, depends upon the pressure and upon the nature of the material—we will call the "starting energy." The "starting energy," therefore, is exactly analogous to the "heat of fusion" and "heat of volatilisation"; also, for brevity, we will call *that temperature at which matter enters this new physical condition the "heat-point,"* and this in its turn is exactly analogous to the "melting-point" and "boiling-point"—marking as it does the temperature at which matter enters a new physical condition. Other conditions being equal, the value of the heat-point will increase with the molecular weight, just as the melting-points and boiling-points do. With hydrogen, for example, it occurs at very low temperatures; but, in the case of the heavy metals, it occurs at a temperature considerably above absolute zero.

To illustrate the theory, we will take the case of a body at normal temperatures and under a very great pressure, but a pressure not sufficiently great to bring the molecules into contact.

Let the body be cooled progressively, keeping meanwhile the pressure constant.

Then ultimately a temperature will be reached, whereat the pressure overcomes the kinetic energy of the molecules and jams them tightly together.

At this point the temperature of the compressed body will fall of its own accord to the absolute zero of temperature, no matter whether the temperature of the surrounding material be above absolute zero or not. Further, if its molecules are all of the same average weight, this change of temperature will take place more or less *suddenly*; *i.e.*, the temperature of the solid will, when a certain temperature be passed, drop more or less abruptly to absolute zero.

The probability of this becomes manifest when we consider the nature of the change. For as compression proceeds at a constant temperature, and as the molecules become crowded more and more closely together, their kinetic energy (the total amount of which must *remain* constant, since the *temperature* remains constant), partakes *less* and *less* of a *translatory*, and more and more of a *rotational* character.

For in a crowded condition any translatory movement on the part of a molecule will immediately bring it into collision with neighbouring molecules, as there is but little play space between them; and the inevitable result of a succession of such collisions will be to gradually convert all the translatory energy of the molecules into rotational energy, provided only we wait long enough.

At the moment before actual molecular contact takes place, we may conceive that all translatory energy has given place to rotational energy.

So that we may liken the molecules at this instant to a vast number of rapidly whirling spinning-tops, almost devoid of translatory movement. The moment these are crowded together, so as to brush against each other as they rotate, enormous frictional forces, due to the molecules grinding one against the other, will start into being and rapidly reduce all to a state of quiescence.

These frictional forces will be all the more marked from the fact that molecules are not smooth round balls, but rather are complicated congeries of atoms, which in their turn have an angular geometrical shape—a statement the truth of which is proved by the science of "stereo-chemistry."

The internal intra-molecular disturbances brought about by the grinding together of the molecular systems will be very great at these low temperatures, and perhaps explains the great display of light and electricity which Dewar has shown to take place when bodies are cooled to a very low temperature, such manifestations being the result *not of molecular* but of *intra-molecular* disturbances.

From the above it is obvious that a body cannot acquire for any length of time *every* temperature; there is in all cases a superior and inferior limit. We may express this by saying that the temperature scale of a body is *not* a continuous function, but vanishes *abruptly* at two points —(*a*) the temperature at which the body decomposes, (*b*) at the *heat-point.* Only for values of temperature intermediate between *a* and *b* is the temperature scale continuous.

University of Berlin, March 26, 1902.

NOTE ON FREE ACID IN SUPERPHOSPHATE.

By J. OSTERSETZER, F.I.C., F.C.S.

Superphosphates, especially those newly manufactured, when being titrated with N/2 or N/10 NaHO solutions in order to determine the free acid present, show beyond the point of opalescence a certain formation of $CaHPO_4$ precipitate, which can be distinctly noticed before the neutral reaction takes place with litmus or paranitrophenol; by using such indicators as dimethylamidoazobenzol, methyl-orange, or alizarin-sulphonic acid, intermediate tints will appear between the point of opalescence and that of neutrality. As regards phosphoric acid, H_3PO_4, Berthelot first observed that on the substitution of the first hydrogen this acid acts as a powerful acid; on the substitution of the second hydrogen as a weaker acid like an organic acid; and on the substitution of the third hydrogen as an alcohol. These different degrees of acidity can be shown by the use of methyl-orange, phenolphthalein, and blue, C_4B, corresponding respectively with the formation of monobasic, dibasic, and tribasic phosphate. It appears, however, that as far as the combination of orthophosphoric acid and lime is concerned, the former may, under given conditions, become to a certain extent loosely attached to CaH_42PO_4, pointing to a possible state of polymerisation of H_3PO_4. It may be interesting to note that by the action of H_3PO_4 upon the rhombic tablets of the tetra-

hydrocalcic phosphate, CaH_42PO_4, at the normal temperature, and the ratio of 6 mols. CaH_42PO_4 to 1 doubled mol. H_3PO_4, a substance can be obtained having all the appearance of one chemicaly combined; the same occurring at 100° C. when 3 mols. CaH_42PO_4 in solution to 1 doubled mol. H_3PO_4 is used.

Staudenmaier (1893) made similar observations by obtaining a compound, KH_52PO_4, corresponding to the doubled molecule of H_3PO_4 by the action of KH_2PO_4 upon H_3PO_4. On the other hand, H. Joulie (1902) shows that there exists a compound, $Na_3H_32PO_4$, intermediate between monosodic and disodic phosphates, the addition of phosphoric acid to Na_2HPO_4 being less than would otherwise be requisite for NaH_2PO_4 (*Comptes Rendus*, vol. cxxxiv., No. 10, March 10, 1902).

When the normal salt Ca_32PO_4 is treated with H_3PO_4 with the object of forming CaH_42PO_4, it may thus be imagined that in the first instance such an apparent excess of phosphoric acid as pointed out above must be used as to counteract the dissociating power of water at 100° C. upon the constitution of tetra-hydrocalcic phosphate; after cooling and on dissolving in water at the ordinary temperature for analytical purposes, a portion of that phosphoric acid will appear as excessive free acid, leaving a residue of the same still loosely combined; the former it is proposed to determine by an intermediate tint of alazarin-sulphonic acid; the change from bright to slightly dark yellow when titrated with NaHO solution coinciding closely with the point of opalescence.

Julius Wagner, in common with Bredig and Winkelbach, attributes the appearance of intermediate tints in the presence of phosphates or other substances to ionisation of the indicators.

The Laboratory,
Messrs. W. and H. M. Goulding, Lim.,
North Wall, Dublin, April 14, 1902.

THE NEW GAS FROM RADIUM.

By E. RUTHERFORD, M.A., D.Sc.,
Macdonald Professor of Physics, McGill University, Montreal,
and
Miss H. T. BROOKS, M.A.

In a recent number of the *Comptes Rendus*, an account was given by Curie of the evidence of the existence of a new gas from radium, which possesses remarkable physical properties. A specimen of very radio-active radium was placed in a glass vessel connected with a mercury pump. On exhausting to a low vacuum and allowing the apparatus to stand, the pressure steadily increased. When the very small volume of gas thus collected flowed along the glass tubes, it rendered them phosphorescent, and if left in for some time, rapidly blackened them. The gas itself was powerfully radio-active, *i.e.*, it continuously gave out a type of Röntgen rays which made gases partial conductors of electricity, and rapidly acted on a photographic plate. This gas preserves its radio-active power for several weeks.

For some time past, one of the authors had been independently investigating one of the most remarkable properties of radio-active substances, namely, the power of continuously emitting radio-active particles of some kind. The term "emanation" was applied to the substance thus emitted, as there was no evidence at the time whether the material emission was a vapour of the substance, a radio-active gas, or particles of matter each containing a large number of molecules.

The "emanation" from thorium compounds was shown to retain its radio-activity for several minutes, and possessed the remarkable property of causing every substance in the neighbourhood of the thorium to become itself radio-active for several days. The specimens of impure radium then in the possession of the author did not possess the power of emitting such an emanation; but Dorn, using a later and more active preparation of radium, showed that it possessed the same emanating power as thorium. One of the most interesting properties of excited radio-activity is that it can be concentrated in an electric field on the kathode, so that a very fine wire of any metal can be made to act like a powerfully radio-active substance for several days.

A short time ago one of the authors published an account of the effect of temperature on the emanating power of radio-active substances, in the *Physikalische Zeitschrift*. In the paper it was shown that the emanating power of thorium increased with rise of temperature to about a red heat, but on heating to a white heat the emanating power was destroyed, and could not be recovered. An examination of a specimen of radium obtained from De Haen, Hanover, showed that the effect of temperature on its emanating power was very large. When the substance was heated below a red heat, its emanating power increased over 10,000 times, but was to a large extent destroyed by heating to a higher temperature. The emanation, obtained by passing a slow current of air over heated radium, was found to preserve its radio-active powers for weeks, when kept in a closed metal vessel. The radio-activity slowly decayed, but was still quite appreciable after a month's interval.

The question now arose, if any physical experiments could be devised to settle the problem as to whether the emanation was in reality a radio-active gas, driven off from the substance, or a vapour of the substance, or a material emission of particles much larger than molecules. Experiments on thorium showed that no appreciable volume of gas could be collected by leaving thorium oxide in a vacuum tube connected with a mercury pump, No new lines were observed in the spectrum of the gas. The amount of the emanation thus given off was thus too small to detect by its volume in this way, but the electrical conductivity produced by the emanation in the gas with which it is mixed is often very large, and can be used as a measure of the amount of emanation present. The emanation gives out a type of radiation which ionises the surrounding gas. When a strong electric field is applied, the current through the gas reaches a maximum value, and is then a measure of the total number of ions produced per second, multiplied by the charge on the ion.

By determining the rate of diffusion of the emanation into air or other gases, using the electrical method, it is possible to obtain an approximate estimate of its molecular weight. The coefficient of inter-diffusion of most of the simple gases have long been known, and the results show the coefficient of diffusion of one gas into another is inversely proportional to the square root of the product of the molecular weights. If therefore the coefficients of diffusion of the emanation into air is found to have a value lying between that of two gases, A and B, we can conclude that the molecular weight of the emanation lies between the molecular weights of A and B.

The apparatus employed was similar to that used by Loschmidt (*Wiener Akad.*, 1871) in his experiments on the coefficient of interdiffusion of gases in the year 1871.

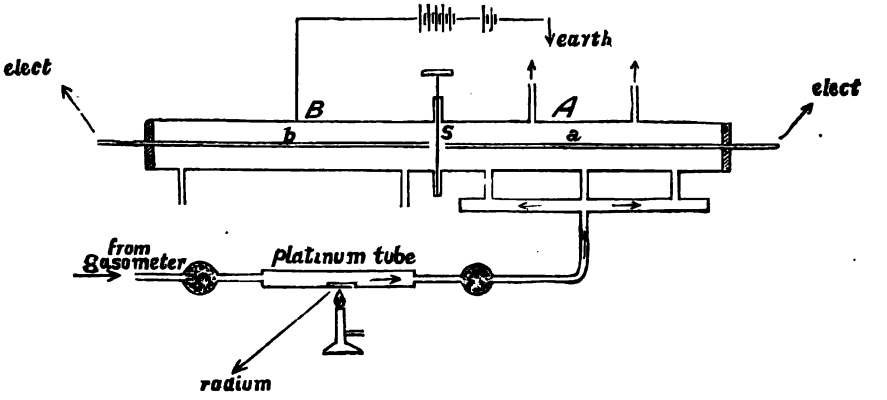
Fig. 1 shows the general arrangement. A long brass cylinder, A B, 6 c.m. in diameter, 73 c.m. long, was divided into two equal parts by a movable metal slide, S. The ends of the cylinder were closed with ebonite stoppers. Two insulated brass rods, *a* and *b*, each half the length of the tube, passed through the ebonite stoppers, and were supported centrally in the tube. The cylinder was insulated and connected to one pole of a battery of 300 volts, the other pole of which was to earth. The central rods could be connected to a sensitive quadrant electrometer.

The cylinder was covered with a thick layer of felt, and placed inside a metal box filled with cotton-wool, in order to keep temperature conditions as steady as possible.

In order to carry a sufficient quantity of emanation into the half cylinder, A, it was necessary to slightly heat the radium. The slide, S, was closed, and the side tubes

opened. A slow current of dry air from a gas bag, passed through a platinum tube, in which a small quantity of a radium compound was placed. The emanation was carried with the air into the cylinder, A. When a sufficient quantity had been introduced, as tested by the electrometer, the current of air was stopped. The side tubes were closed by fine capillary tubes. These prevented any appreciable loss of gas due to diffusion, but served to keep pressure of gas inside A at pressure of outside air. The three entrance tubes into the cylinder, shown in the figure, were for the purpose of initially mixing the emanation and gas as uniformly as possible.

After standing for several hours to make temperature conditions steady, the slide was opened, and the emanation began to diffuse into the tube B.



The current through the tubes A and B was measured by an electrometer, with suitable capacity in parallel, at regular intervals. Initially there is no current in B, but after the opening of the slide, the amount in A decreased and the amount in B steadily increased. After several hours the amount in each half is nearly the same, showing that the emanation is nearly uniformly diffused throughout the cylinder.

It can be readily shown that if—

K = coefficient of diffusion of the emanation into air.
f = duration of diffusion experiments in secs.
a = total length of cylinder.
S = amount of emanation in tube A at end of diffusion.
S_2 = amount of emanation in tube B at end of diffusion, then—

$$\frac{S_1 - S_2}{S_1 + S_2} = \frac{8}{\pi^2}\left\{ e^{\frac{-\pi^2 K f}{a^2}} + \tfrac{1}{9} e^{\frac{-\pi^2 K f}{a^2}} + \&c. \right\}$$

(See Stefan and Loschmidt, *Berichte Wien. Akad.*, lxiii., 1871).

From this equation K can be determined, if S_1 and S_2 are known.

An uncertainty, however, arises, in estimating S_1 and S_2 for the rate of leak in A and B is made up of the current due to emanation alone, and the current produced in the gas by the excited radio-activity on the electrodes. As the amount of excited radio-activity increases with the time, the ratio of the current due to the emanation and to the excited radiation varies with the time allowed for diffusion. The ratio of the current due to the excited radiation can be determined by removing the central electrode, and finding the amount of current immediately after the introduction of a new electrode.

When the emanation is allowed to diffuse for half-an-hour, the current due to excited radio-activity was about 0·4 of the whole.

The calculated value of K was found to be about 20 per cent greater when the correction for the amount of excited radio-activity was applied.

The value of K deduced from the experiments was found to be between 0·08 and 0·15. All the later observations gave a value about 0·08.

This variation in the value of K deduced from the experiments is not altogether due to errors of experiment, the values obtained at first with a new specimen of radium were in all cases higher than when it had been laid by for several months. It appears as if the emanation were not simple in character, and that part of the emanation first given off was of lower molecular weight than that emitted after several months' exposure to the air. Further experiments are now being carried out to see if the radium emanation undergoes a progressive change with time. For the purpose of comparison, we will now give a few of the coefficients of interdiffusion of gases, compiled from Landolt and Bernstein's tables:—

Gas or vapour.	Coefficient of diffusion into air.	Molecular weight.
Water vapour	0·198	18
Carbonic acid gas	0·142	44
Alcohol	0·101	46
Ether	0 077	74

In the above table we see that the coefficient of interdiffusion follows the inverse order of the molecular weights. In cases of the simpler gases it has been shown experimentally that the coefficient of interdiffusion is approximately inversely proportional to the square root of the product of the molecular weight. If we apply these considerations to the emanations (K = 0·08 to 0·15) we see that it is a gas or a vapour of molecular weight (allowing a wide margin) probably lying between 40 and 100. These numbers exclude the possibility of the substance being a vapour of radium, for it has already been shown by M. and Mdme. Curie that the atomic weight of radium is greater than that of barium. We must therefore conclude that the emanation is in reality a heavy radio-active vapour or gas.

On account of the rapid decay of the radiating power of thorium emanations, it is not possible to determine its coefficients of diffusion in the same way; but special experiments show that it diffuses rapidly, and is also probably gaseous in character. The physical properties of these emanations or gases are most remarkable. The radium emanation not only continues for long intervals to be a source of radiation which is apparently similar in character to easily absorbed Röntgen rays, but in some way manufactures from itself a positively charged substance, which travels to the negative electrode and becomes a source of secondary radio-activity.—*Transactions of the Royal Society of Canada*, Series 2, vii., p. 21.

ESTIMATION OF TITANIUM.

By GEO. B. WATERHOUSE,
Bursar of the Royal Commissioners, Exhibition of 1851.

TITANIUM is an element that seems to have offered great difficulty to steel works' chemists. Although many processes have been submitted, there has not yet been one sufficiently short and accurate to be applied to working-day uses. It is hoped that the following process will provide for this need.

In looking over the various gravimetric methods suggested, it is at once seen that three things are common to almost all:—

1. Precipitation, by boiling, from largely diluted solutions.
2. Fusion with sodium or potassium carbonate.
3. Fusion with acid potassium sulphate.

Precipitation by simply long-continued boiling from solutions is, however, seldom complete, and the precipitate is contaminated with aluminium, silicon, phosphorus, and ferric salts, if these are present. The boiling also occupies a considerable time, and the precipitate is very liable to stick pertinaciously to the sides of the beaker. Boiling, then, in this way is an undesirable method of precipitating if another can be found. Titanium may be precipitated from solutions by ammonium hydrate—a fact made use of by Tosh (CHEMICAL NEWS, vol. xvi., p. 168), by Moissan (CHEMICAL NEWS, vol. lxxi., p. 103), and by others. But for the precipitate to be pure the titanium in solution must be separated completely from iron, aluminium, and other elements.

But, by boiling the solution, having previously eliminated all free acid, except about 1 per cent, with sodium acetate and 10 to 15 per cent glacial acetic acid, the whole of the titanium is precipitated after a brisk and short boiling, along with any silica and phosphorus present. Ferric salts must be removed, as they hinder the precipitation.

Fusion with sodium or potassium carbonate does not, as is stated, always render the whole of the silica soluble, but often along with the titanium, which is all insoluble, some of the silica is found. Phosphorus, however, is completely soluble as sodium or potassium phosphate.

Some processes, as Morgan's (CHEMICAL NEWS, vol. lxxv., p. 134), rely on the assumption that titanium in acid solutions (with the presence of sufficient phosphoric acid) is all rendered insoluble after baking as a phospho-titanate, but often some is still soluble, which is, of course, unaccounted for.

Gravimetric Estimation of Titanium in Steels or Pig-irons.

Weigh out 5 grms. of drillings into a 20-oz. beaker. Add 50 c.c. of strong hydrochloric acid solution. Heat gently until the steel is dissolved; then boil briskly and evaporate to dryness; bake, with a strong final heating. Remove from the plate, cool, and add enough strong hydrochloric to thoroughly saturate the dry chlorides, about 20 c.c. Heat for a few minutes, then add about 70 c.c. water, and boil until all the chloride of iron is dissolved. Filter through a small paper, and wash until entirely free from iron with a little warm 1 : 1 hydrochloric and cold water. Save the precipitate.

Place the filtrate in a 20-oz. beaker, make the volume up to about 150 c.c.; without heating add, little by little, dilute liquid ammonia until the acid is neutralised and there is a faint precipitate. Clear this precipitate with a few drops of hydrochloric; then add slowly about 50 c.c. of a 1 : 5 sodium sulphite solution. If a precipitate forms, or a dark colour persists, add a little dilute sulphuric acid until it is removed.

When the iron is in the ferrous condition, as seen by the colour, heat, and as it begins to boil add 50 c.c. glacial acetic acid and a filtered solution of about 20 grms. pure sodic acetate which have been heated in a separate beaker. Boil briskly for fifteen minutes.

If necessary, allow any precipitate to settle a little, and filter through a large double paper. Wash the precipitate well out of the beaker, and wash the paper well with hot water. Dry this and the previous precipitate.

When dry, ignite them in a deep 3-inch platinum dish until all graphite and filter-paper are burned away. Place this dish in a desiccator. When cool, intimately mix the precipitate with about 10 grms. of pure dry powdered sodium carbonate. Fuse the mixture in the covered dish over a gas blowpipe until it is quite liquid, at which condition it is maintained for several minutes.

When cold, the fusion is thoroughly extracted in about 150 c.c. hot water. The dish is washed and removed. Allow the precipitate to settle, and filter through a hardened paper. The beaker and precipitate are washed very well with hot water.

Cautiously spread out the filter-paper on a clock-glass, and wash the precipitate into the beaker. Any adhering precipitate is dissolved off by means of about 10 c.c. hydrochloric acid, heated in the platinum dish, and poured over the paper. Wash all the yellow tinge out of the paper. To the beaker containing the precipitate, acid, and washings, add about 10 c.c. dilute sulphuric acid.

Cover the beaker and boil briskly down until white SO_3 fumes are seen. Remove from the plate, cool, and dilute to about 50 c.c. Filter if necessary, washing the paper well.

Dilute the filtrate to about 150 c.c. Add dilute ammonia until a faint permanent precipitate is obtained. Re-dissolve this as before, but only add 20 c.c. of the sodium sulphite solution. When colourless, heat the solution to boiling and add sodic acetate and acetic acid as before. Boil well for fifteen minutes. Allow the precipitate to settle, and filter through a large double paper. Wash the paper well round the edges with hot water. Dry as usual, and ignite in a weighed platinum crucible. The increase in weight is TiO_2 containing 60·97 per cent titanium.

To verify the above process, a standard solution of pure TiO_2 was made in the following way:—

Reputed pure titanic acid was taken, ignited strongly, and fused with an excess of sodium carbonate. The fusion was extracted, the precipitate filtered off, dissolved in hydrochloric and sulphuric, and evaporated to low bulk to separate SiO_2. The titanium in the filtrate was precipitated by boiling with sodic acetate and acetic, filtered, washed, and ignited.

One grm. of the pure TiO_2 was then taken, fused with an excess of sodium carbonate, and the fusion extracted in sulphuric acid, about 2 : 1. The dish was washed and removed, and the clear solution transferred to a 120 c.c. flask and diluted to the mark.

Known quantities of this solution were introduced to different weights of pure iron, and the following results obtained:—

Weight of iron taken. Grms.	C.c. added.	Theoretical weight of precipitate. Grm.	Weight obtained.	Theoretical percentage.	Percentage obtained.
5	1·0	0·0083	0·0084, 0·0086	0·1016	0·102, 0·103
4	2·5	0·0207	0·0209, 0·0212	0·317	0·318, 0·323
5	6·0	0·0500	0·0501, 0·0508	0·6097	0·610, 0·615
2	4·1	0·0341	0·0346	1·041	1·064
1	4·0	0·0333	0·0334	2·020	2·036

As it was also desirable to judge the influence of other elements on the process, a mean of 1 per cent of the following elements was introduced in such a form that they could go into solution, and the following results obtained:—

Weight of iron taken. Grms.	C.c. added.	Percentage of foreign elements.	Theoretical percentage of titanium.	Percentage obtained.
2	4·0	1 per cent Cr	1·016	0·980
2	4·0	1 ,, Al	1·016	1·018
2	4·0	1 ,, Ni	1·016	0·994
2	4·0	1·1 ,, W	1·016	1·012
2	4·0	1·2 ,, Mo	1·016	1·001
2	4·0	1·1 ,, V	1·016	1·048

Though the last result is a little high, the theoretical and practical weights of precipitate are very near, *i.e.*,—

Theoretical .. .. 0·0333 grm.
Weight found .. .. 0·0345 „

As a check on the solution, 4 c.c. were taken and the Ti precipitated with dilute ammonia, filtered off, washed, and weighed after ignition:—

Theoretical weight .. 0·0333 grm.
Weight found .. .. 0·033 „

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING MARCH 31ST, 1902.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To CHARLES PERRIN, Esq., M.Inst.C.E.,
Water Examiner, Metropolis Water Act, 1871.

London, April 10th, 1902.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 192 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from March 1st to March 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 192 samples examined by us during the month, all were found to be clear, bright, and well filtered.

The deficit of rain still continues; the amount measured at Oxford during the month was 1·20 inches, the average fall is 1·47 inches; this makes a deficit of 0·27 inch, which added to the previous one of 2·27 inches gives a total deficit for the first three months of this year of 2·54 inches, or 47·4 per cent on the thirty-five years' average.

Our bacteriological examinations of 484 samples have given the results recorded in the following table; we have also examined 40 other samples, from special standpipes, wells, &c., making a total of 524 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 22 samples) ..	336
New River, filtered (mean of 116 samples) ..	13
Thames, unfiltered (mean of 24 samples) ..	2013
Thames-derived water, from the clear-water wells of eight Thames-derived supplies (mean of 268 samples)	28
Ditto ditto .. .. highest	292
Ditto ditto .. .. lowest	0
River Lea, unfiltered (mean of 22 samples) ..	710
River Lea, from the East London Company's clear-water wells (mean of 31 samples) ..	12

Of the 415 samples of impounded and filtered water supplied to London and examined bacteriologically during the month, 20 samples, or 4·8 per cent, were sterile. Eight samples contained more than 150 microbes, while seventeen samples, or 4·0 per cent, contained more than 100 microbes per c.c. The mean number of microbes in the seventeen excess samples was 166, against a corresponding mean of 143 in nine excess samples during February. Thus it appears that the mean bacterial quality is not quite so high as it was last month. This must not be interpreted, however, as inferring that the majority of the London Companies were distributing less effectively filtered water; because a temporary falling off in the efficiency of a filter bed by any one Company is sufficient to increase the average of the whole, so as to produce a deteriorated mean result, such as we see this month on comparing the figures with those of last month.

We are, Sir,
Your obedient Servants,
WILLIAM CROOKES.
JAMES DEWAR.

ON THE

USE OF SALICYLATE OF SODIUM FOR THE ESTIMATION OF MIXTURES OF TERPENIC ALCOHOLS AND OF THEIR ETHERS.

By G. DARZENS and P. ARMINGEAT.

IN the October number of the *Bulletin Scientifique et Industriel*, MM. Charabot and Hébert published a paper entitled "Researches on the Mechanism of Etherification in Plants," in which they described a very simple method for the estimation of a mixture of terpenic alcohols and ethers.

This method depends on the solubility of these alcohols in a 50 per cent solution of salicylate of sodium, which dissolves neither their ethers nor their terpenes.

We were ourselves at that time occupied with this curious property of the salicylate, more especially with regard to the commercial purification of terpenic alcohols; but we also perceived that the simplicity of this process was only deceiving, and that it did not lead even to an *approximate* result.

We think it worth while to publish our results, as the proposed method of analysis can only lead to grave misconceptions if used without due warning.

We operated on mixtures of linalol, geraniol, rhodinol, and their acetic ethers, first making a mixture of one of these alcohols with its acetic ether, a mixture of which we accurately determined the coefficient of saponification, by which we were able to tell its proportion of ether. We then treated this mixture with four times its volume of 50 per cent salicylate of sodium; after twenty-four hours, the insoluble portion was decanted, and the solution of salicylate was precipitated by an excess of water, to recover the part dissolved. We then determined the coefficient of saponification of each of these portions.

We found that in every case the part dissolved contained large proportions of ether, as can be seen in the following table, which gives a summary of our experiments:—

Mixtures.			Part insoluble in the 50 per cent salicylate of sodium.		Part soluble in the 50 per cent salicylate of sodium.	
Nature of the mixtures.	Coeff. of saponification.	Ether per cent.	Coeff. of saponification.	Ether per cent.	Coeff. of saponification.	Ether per cent.
Geraniol and acetate of geranyl ..	116	40·6	110	38·5	120·3	42·3
Linanol and acetate of linalyl ..	98·2	34·4	116·3	40·7	85	29
Rhodinol and acetate of rhodinyl ..	126	45	The mixture was completely soluble.			

This result, which is rather deceiving, should not cause any surprise, as it agrees very well with our knowledge

of the properties of solutions. The following is the explanation:—

The solubility of the terpenic alcohols in salicylate of sodium at 50 per cent is not a chemical phenomenon, but simply a physical one; an occurrence which is better accounted for by phenomena of capillarity and thermodynamics than by chemical combination.

Thus we see that *ether being miscible, in all proportions, in its generating terpenic alcohol*, should also dissolve in the alcoholic solution in the salicylate; this solution being only a simple physical mixture must play a part in the properties of its constituents.

To prevent there being any doubt remaining as to this solubility of the ethers, and the interpretation we have put upon it, we have confirmed it directly by the following experiment:—

A solution of 10 c.c. of rhodinol in 40 c.c. of 50 per cent salicylate of sodium was prepared; then, by means of a graduated burette, acetate of rhodinyl was added; this dissolved *instantly*.

By operating carefully and slowly, we were able to dissolve as much as 20 c.c. of acetic ether.

A moment, however, arrives when one drop of ether separates the mixture into two layers, as M. Duclaux observed with his mixtures. This limit of proportion is a function of the temperature at which the experiment is carried out, which further complicates the matter.

What we have stated with regard to mixtures of alcohols and ethers, also applies to mixtures of alcohols and terpenes.

A direct experiment finally showed us that we cannot depend on the salicylate to extract the terpenic alcohols from a natural essence containing alcohols.

An essence of geranium from Bourbon, having a coefficient of saponification of 72·9, was shaken up with four times its volume of a 50 per cent solution of salicylate of sodium. The recovered soluble portion had a coefficient of saponification of 64·2.

In publishing these remarks we in no way contradict the conclusions arrived at by MM. Charabot and Hébert; it would be bold to assert that the salicylate of sodium process could never be of use in experienced hands when warned of the danger, and in certain cases where precision is not of great consequence it might be of considerable service.—*Bull. Soc. Chim.*, Series 3, vol. xxv., No. 24.

THE CEMENTATION OF IRON BY SILICON.

By P. LEBEAU.

In his experiments on silicide of iron, $SiFe_2$, M. Moissan observed that the combination of silicon with iron could be effected before the fusion of either one or the other of these elements. He heated cyclinders of soft iron packed in crystals of silicon, at the temperature of a good forge. Under these conditions the metalloid was not fused; however, the cylinders of iron, although they kept their shape, were transformed into silicide of iron, even to the axis of the cylinder. They contained 2 per cent of combined silicon.

We have recently had occasion to observe a similar fact, but one produced at much lower temperatures, and which causes this action of silicon on iron to appear very like the cementation of the same metal by carbon.

If we heat an intimate mixture of reduced iron and very finely divided silicon to 950° *in vacuo*, or in a current of hydrogen, the combination between these two metals takes place at a temperature very far from that of their fusing-points.

The mixture of iron and silicon, which was compressed into the form of small cylinders, was found not to have changed its shape; it had acquired a greater solidity, but, as before, presented a spongy appearance and a dull tarnished fracture. When treated with nitric or hydrochloric acid, it dissolved without leaving any residue; all the silicon had entered into combination.

With a view to ascertaining whether this facility of combination could be attributed only to the state of division of the materials employed, we heated under the same conditions a small parallelopiped of soft iron, one of whose faces was polished and on which we simply laid a few crystals of silicon. After heating for five or six hours at the same temperature, we examined this polished surface. We were able to see with the naked eye that each crystal had left a trace of the image of its face adhering to the iron; the contours were slightly shadowy.

Similar facts were observed with nickel and cobalt.

We propose to examine this cementation of iron and other metals by silicon in a more thorough manner, notably to see if the presence of certain gases, such as nitrogen or carbonic oxide, would not influence the action.

Further, we may be able to apply it to the metallographic study of the metallic compounds of silicon.—*Bull. Soc. Chim.*, Series 3, vol. xxvii., No. 3.

THE ACTION OF SULPHURETTED HYDROGEN ON BROMIDE OF BORON.

By A. STOCK and O. POPPENBERG.

Sulphuretted hydrogen does not act on chloride of boron in the cold; the action only commences on the chloride of boron fumes at a temperature above 250°, and at a dull red heat a certain amount of impure sulphide of boron is formed, contaminated with chloride. Thermochemical facts enable us to foresee a better result with bromide of boron, and, in fact, we find that the reaction takes place in the cold, giving a white body in the form of beautiful crystals. As these latter permeate the whole of the liquid and interfere with the end of the reaction, it is found to be more convenient to act with the sulphuretted hydrogen on a solution of bromide of boron in benzene or sulphide of carbon. The authors carried out their experiments in a small flask with a glass tube ground in with emery; the parts are fitted together and arranged so that the lower part can be easily removed if it becomes obstructed with a deposit of crystals, and also so that the bromide of boron and the solvent, when volatilised, can be condensed in a vertical condenser, and run back into the flask without interfering with the flow of sulphuretted hydrogen. The end of the escape tube is fitted with a drying tube filled with chloride of calcium. Fifty grms. of bromide of boron were dissolved in the same weight of sulphide of carbon, and well-dried sulphuretted hydrogen was passed through slowly.

In a short time torrents of HBr were given off; after forty-eight hours these fumes had almost ceased to appear. The action of the sulphuretted hydrogen was continued for six days and nights, when the operation was stopped and the liquid evaporated to dryness *in vacuo*. A well-crystallised body was then formed, which was re-crystallised in benzene or sulphide of carbon. This substance is a boric sulphydrate, $Bo_2S_3.H_2S$ or $Bo_2S_4H_2$, corresponding to metaboric acid; it crystallises in long white needles having the odour of sulphuretted hydrogen; it is quickly decomposed by water into BoO_3H_3 and H_2S; soluble without decomposition, and re-crystallisable in benzene and sulphide of carbon (1 part in 5 of warm benzene, or sulphide of carbon, but very slightly soluble in this latter at −20°). It also reacts on alcohol, giving H_2S, and it appears to combine with ether; these reactions will be examined later on. By the action of heat at the ordinary temperature, this body loses sulphuretted hydrogen, the crystals effloresce and turn yellow, leaving sulphide of boron; this reaction is noticeable at 100°, and is complete at 300°. It is also preferable to work with sulphide of carbon rather than with benzenic solutions, as these latter, if heated to boiling-point, commence to give off a

little sulphide of boron. But in sealed tubes the sulphydrate melts at 120°; at 140° we obtain a clear colourless liquid, which on cooling takes the form of a white crystalline mass, and is recovered unaltered. For the purpose of analysis this substance was treated with potash solution, the sulphur estimated in the form of Ag_2S, and the boron by polarimetry in the presence of mannite. Cryoscopic measurements of the benzenic solutions give the formula $Bo_2S_4H_2$ and not BoS_2H.—*Berichte*, vol. xxxiv., p. 399.

A NEW INVESTIGATION CONCERNING THE ATOMIC WEIGHT OF URANIUM.*

By THEODORE WILLIAM RICHARDS
and
BENJAMIN SHORES MERIGOLD.

(Continued from p. 188).

The following method was found to fulfil the required conditions fairly well:—A quantity of the iodate was placed in a boat in a combustion tube, to one end of which was attached, by a ground glass joint, a weighed U-shaped tube. The free end of this tube was drawn out and fused to a smaller tube, which dipped into a solution of sulphurous acid. On heating the iodate in a stream of air and oxygen, the salt was decomposed, and the iodine was carried over and condensed in the U-tube, which was packed in ice. The small quantity of iodine vapour not condensed was collected in the sulphurous acid, and precipitated as silver iodide. The heating was continued for an hour after no more iodine could be seen coming off. The end of the U-tube was then sealed by fusing off the small tube, and the other end was closed by a ground glass stopper immediately after disconnecting from the combustion tube. In this way about 99 per cent of the total iodine was weighed directly as free iodine. Of course the small amount of iodine remaining in the oxide after ignition had to be determined separately, as already described. By this method the amount of iodine found was practically identical with that found by the sulphurous acid method.

In determining the iodine present in the oxide after ignition, it has been assumed that the iodine is present as iodide. Although it is hard to believe that at the temperature employed any of the iodine can exist as iodic acid, it is impossible to prove the point experimentally. The uncertainty in regard to this point renders the use of the method inadvisable where the greatest possible accuracy is desired. Hence none of these analyses have any significance as a basis for computing the atomic weight of uranium.

Besides the bright yellow, slightly soluble iodate, we prepared a paler yellow, more soluble, and more highly hydrated salt, which suffers transition quickly into the earlier compound at a high temperature, and more slowly at a low temperature. Double iodates with sodium and potassium were also prepared. Some of our observations were inconsistent with the published record concerning the subject; but in spite of our desire to clear up the uncertainty and to study the rather interesting transition phenomena, we abandoned the iodates because none of them gave promise of a precise basis for the determination of the desired atomic weight.

The next compound investigated was the oxalate, which has the composition $UO_2C_2O_4.3H_2O$. Owing to the comparatively slight solubility of this compound it can be obtained in a state of great purity by a few crystallisations.

The best method of analysis is that of dry combustion, the carbon dioxide being absorbed in potash in the usual manner. The uranium is left in the combustion tube as the green oxide, U_3O_8, and, consequently, can be compared directly with the weight of carbon dioxide obtained. This obviates the necessity of using the weight of the oxalate as a factor in the calculation of the atomic weight, and so eliminates the error due to included water. As already mentioned, this method has been used by Ebelmen and Péligot in their determination of the atomic weight of uranium. There is in this method a possible source of error, difficult of detection and correction, but none the less dangerous, in the possibility that the uranium oxide may after combustion still retain traces of carbon. Moreover, it became evident, after a few analyses had been made, that combustion analysis, as ordinarily conducted, is an exceedingly questionable method where great accuracy is desired. The great difficulty in obtaining absolute "blanks" is well known. Our experience amply confirmed the observations of Mabery ("Inaccuracies in the Determinations of Carbon and Hydrogen of Combustion," *Journ. Amer. Chem. Soc.*, 1898, xx., 510), Auchy (*Journ. Amer. Chem. Soc.*, 1898, xx., 243), and others in regard to the loss of water and possibly of carbon dioxide from the ordinary form of potash bulbs. We also found a single sulphuric acid tube entirely insufficient to absorb all the water. Clearly, then, if we were to use this method, an elaborate investigation of the form of apparatus, method of procedure, and limits of error, was absolutely imperative. The use of the oxalate, however, did not seem sufficiently promising to warrant the necessary expenditure of time.

After thus investigating the uranium compounds which seemed likely to furnish a suitable basis for an atomic weight determination, anhydrous uranous bromide, in spite of its disadvantages, seemed most likely to fulfil the necessary requirements. As already mentioned, this compound oxidises with the greatest ease on exposure to moist air. It was necessary, therefore, to devise apparatus which should preclude any possibility of bringing the sublimed bromide in contact with the air of the laboratory until it had been collected an weighed. After much experimenting with different forms of apparatus, the following method was adopted.

(To be continued).

* Contributions from the Chemical Laboratory of Harvard College. From the *Proceedings of the American Academy of Arts and Sciences*, xxxvii., No. 14.

NOTICES OF BOOKS.

Engineering Chemistry; a Practical Treatise for the Use of Analytical Chemists, Engineers, Iron-masters, Iron-founders, Students, and others. By H. Joshua Phillips, F.I.C., F.C.S., &c. Third Edition, Revised and Enlarged. London: Crosby Lockwood and Son. 1902. Pp. 406.

The second edition of this book, published in 1894, now being out of print, it has been found necessary to publish this the third edition, and the opportunity has been seized to make several important additions to both the text and the illustrations. Compared with the first edition of 1891 the work has been increased by more than 120 pages and about 30 new illustrations, while the contents have again been brought up-to-date.

The principal addition to the present issue is the fully illustrated section upon the microscopical examination of iron and steel, for the substance of which the author acknowledges his indebtedness to Mr. J. E. Stead, F.I.C. Other additions include particulars of Hampe's method for the estimation of oxygen in copper, Hehner's bromine thermal test for oils, the Pensky-Marten flash-point apparatus, Redwood's apparatus for testing air for inflammable vapour; the fluidimeter, an instrument designed by the author for ascertaining the fluidity or viscosity of several oils at the same time; the Home Office test for the determination of the stability of explosives, &c. These additions have considerably added to the value of the book, which we believe will be received with as much favour as the previous editions have been.

Primary Batteries; Their Theory, Construction, and Use. By W. R. Cooper, M.A., B.Sc. London: The *Electrician* Printing and Publishing Company, L'd. Pp. 324.

Primary batteries are, to say the least of it, disappointing; they have raised hopes of great possibilities, but the realisation of these hopes has been comparatively small. Two of the great drawbacks to them are that they are generally expensive and nearly always "messy." However, they have their uses, and the advance made in recent years is by no means small, and most readers will be surprised on looking through these pages at the large number of primary batteries that there really are.

Many important additions have been made of late to the theory of the voltaic cell, and a considerable number of pages have been devoted to this part of the subject.

With regard to the practical part of the subject the author's object has been to describe those batteries which are in general use, or of particular theoretical interest, rather than to include all known, or unknown, batteries whether they are good, bad, or indifferent.

A specially interesting and valuable feature of this book is the comparison of the various kinds of batteries, showing their capabilities, and the author has included a number of curves obtained experimentally during the past few years, and which enable such comparisons to be made.

The book is divided into twelve chapters; the earlier chapters deal with the simple voltaic element, local action, and polarisation (a very comforting word, which is often used to hide ignorance), and the theory of the voltaic cell. We then have an interesting chapter on non-chemical cells and thermopiles, followed by a practical chapter on testing cells.

The last five chapters are devoted each to a particular kind of battery, viz., one and two fluid cells, dry cells, standard cells, and carbon consuming cells. Owing to the importance of the subject, standard cells have been dealt with at some length. The first really successful standard cell was that introduced by Latimer Clark in 1873, and this, after certain modifications as to the treatment of the materials, was finally given a legal position in this country in 1894, and is now known as the Board of Trade Clarke Cell.

Chapter XII., on carbon consuming cells, and the commercial generation of electrical energy, is of interest, and the subject may possibly be of great importance in the future. One of the first difficulties to be contended with in this class of battery is that of getting the carbon into solution, and it is necessary that solution should take place by means of ions, and that the solution obtained should be an electrolyte. Unfortunately, carbon does not form salts, and when used as an anode in hydrochloric acid, although a chloride CCl_4 is formed, this product has none of the properties of a salt, and is, moreover, an insulator. The various attempts which have been made to solve this problem are interesting, but at the same time discouraging, and the difficulties to be overcome appear to preclude the possibility of commercial success, at least in the near future.

The Tutorial Physics. Volume IV. A Text-book of Magnetism and Electricity, with 170 Illustrations, and Numerous Examples. By R. Wallace Stewart, D.Sc. (Lond.). Fifth Edition. London W. B. Clive, University Tutorial Press. 1902. Pp. 368.

This, the fifth edition of this book, follows closely on the fourth edition, which was published in 1899. The arrangement originally adopted remains unchanged; the book is divided into three parts, viz.:—Part I., Electrostatics; Part II., Magnetism; and Part III., Current Electricity; comprising eighteen chapters in all, with a further thirty-five pages of appendices, &c.

These publications are well known by now, and afford a great deal of help to students and candidates for the matriculation, intermediate science, and preliminary scientific examinations of the University of London, and their appreciation of them is apparent.

CORRESPONDENCE.

ARSENITE OF SILVER.

To the Editor of the Chemical News.

Sir,—Your readers will all be glad to see Mr. J. A. Wanklyn's name again as that of a contributor to your columns. To me his communication (p. 181) has a special and personal interest, as proving that my remarkable sleep for a long term of years has an exact parallel in the experience of Mr. Wanklyn.

Mr. Wanklyn's contribution commences with the statement that the result of an analysis of the canary-yellow arsenite of silver, made recently in his laboratory, "astonished" him. His surprise was due to the fact that the analysis showed the compound to contain Ag_3: As instead of Ag_3: As, as Mr. Wanklyn expected.

It is true that the well-known yellow arsenite of silver is described as "the tetrargentic salt, $2Ag_2O,As_2O_3$" in vol. i. of "Watts' Dictionary of Chemistry," published in 1866. On the other hand, in the First Supplement to that work, published in 1872, the formula is given as Ag_3AsO_3, and this is true also of Morley and Muir's edition, published in 1888.

I have consulted a number of "chemical text-books" published at different periods, and have met with no instance since 1861 in which the obsolete formula so long cherished by Mr. Wanklyn has been adopted. Even the English edition of "Gmelin," published in 1852, gives $3AgO,AsO_5$ as the formula of the yellow precipitate produced on adding ammonio-nitrate of silver to a solution of arsenious oxide. Among the text-books giving Ag_3: As are Miller, 1863; Noad, 1864; Bloxam, 1867; Thorpe, 1873; Roscoe and Schorlemmer, 1877; Valentin and Hodgkinson, 1880; Attfield, 1881; Haughton Gill, 1883; Prescott and Johnson, 1890; &c.

Mr. Wanklyn's observation was anticipated forty years since by Charles L. Bloxam, who, in a paper published in the *Journal of the Chemical Society* for 1862 confirmed Filhol's formula, Ag_3AsO_3, and suggested the contamination of the precipitate with excess of arsenious acid as the probable cause of the erroneous formula attributed to the compound by Pasteur.—I am, &c.,

Rip van Winkle.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

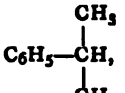
Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xxv., Nos. 18—19.

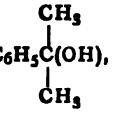
The Phenomena of Combustion in Industrial Furnaces.—O. Boudouard.—The author has already given the numerical results he obtained from the examination of the reaction $2CO \rightleftarrows CO_2+C$. He now shows how these facts enable him to definitely establish the theory of the different commercial operations, which have been based up to the present more or less on conjecture. The reaction $CO_2+C=2CO$ is a function of several important factors, and the best conditions for obtaining practical results are—(1) To have as high a temperature as possible, (2) the fuel must be very porous and finely divided, and (3) the speed of the current of gas should be as slow as possible, though if too slow the contact between the gas and the carbon will be too prolonged.

Estimation of Platinum and Iridium in Platinum Ores.—MM. Leidié and Quennessen.—Already inserted in full.

The Oxidation of the Homologues of Benzene.—Eyvind Boedtker.—The author undertook to show whether the careful oxidation of isopropylbenzene,—



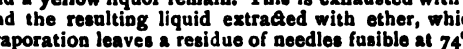
would give a phenyldimethylcarbinol,—



as expected by M. Friedel, and he finds that it is not only the group CH that is susceptible of fixing an atom of oxygen, but that the primary group CH_3 has the same property and apparently to the same degree.

The Electrolytic Oxidation of the Nitrotoluenes.—P. Pierron.—The electrolysis of orthonitrotoluene is conducted in a beaker heated on the water-bath and containing a porous pot of 150 c.c. capacity. The anode, formed of a strip of platinum, is placed inside, while a strip of nickel on the outside forms the cathode, plunged in sulphuric acid at 75 per cent. The mixture to be oxidised is placed in the porous pot, and the whole heated to 90° and electrolysed with a current of 1 ampère for fifteen hours. The acetic acid and the nitrotoluene of the mixture are carried off with the steam, and a tarry residue and a yellow liquor remain. This is exhausted with water, and the resulting liquid extracted with ether, which, on evaporation leaves a residue of needles fusible at 74°. The acetylised derivative is orthonitrobenzylic alcohol.

Paramethoxyatrolactic Acid,—



—J. Bougault.—Five grms. of *p*-methoxyhydratropic acid are dissolved in 100 c.c. of water and treated with 12 c.c. of soda-lye. A solution is also made of 25 grms. of $KMnO_4$ in 500 c.c. of water and 25 c.c. of soda lye. The first solution is kept in a water-bath at 15—18°, and the $KMnO_4$ solution added by 100 c.c. at a time, about once an hour; the temperature must not be allowed to exceed 20°. It is then left for twelve hours after the last addition of permanganate, when it is found to have changed to a green colour. The manganate is destroyed by a slight excess of bisulphite of sodium and filtered. The solutions are evaporated down to about 100 c.c., neutralised with hydrochloric acid, and treated with a small excess of chloride of zinc. Paramethoxyatrolactate of zinc is precipitated; this is collected and decomposed with dilute hydrochloric acid in the presence of ether. This latter, washed and evaporated, leaves the pure—or practically pure—acid. This acid is anhydrous, is slightly soluble in cold water, easily soluble in boiling water, alcohol, and ether; it melts at 129—130°; it decomposes slowly at 100°.

Methylene-3 . 4-Dioxyhydratropic Aldehyde and Acid.—J. Bougault.—This aldehyde is a colourless liquid, odourless when cold, but giving off very pungent fumes when heated. It is almost insoluble in water, but soluble in alcohol, ether, and chloroform. Its density at 15° is 1·203. It boils at 279—280°, and can be distilled at the ordinary pressure without decomposition. The acid is prepared by oxidising the aldehyde by means of oxide of silver in an alkaline solution. It forms anhydrous prismatic crystals, and fuses at 80°; its isomer, methylenehydrocafeic acid, fuses at 84°. A solution of 1 in 500 of this acid, exactly neutralised with soda, forms salts when precipitated with nitrate of silver, sulphate of copper, mercurous nitrate, and perchloride of iron; in the latter case the precipitate is re-dissolved on heating, and forms a red solution. The properties of these salts are described.

Phenylcarbazinate of Phenylhydrazine.—P. Freundler.—This substance is formed when the base is in the presence of an excess of CO_2. The return depends essentially on the concentration, but cannot exceed 65 per cent of theory.

	Quantity used. Grms.	Vol. of CO_2. C.c.	Wt. of dry carbazinate. Grms.
I.	20	300	6·5
II.	20	600	10
III.	20	1200	9
IV.	20	500	16

The theoretical return is 24 grms. It occurs in the form of a white crystalline powder, slightly soluble in cold water, decomposed by hot water and alcohol; it fuses at about 80°.

The Sulphonised Oxyazoïc Colouring-matters and their Salts.—P. Sisley.—A long paper, not suitable for abstraction.

Hydrocinchonine.—E. Jungfleisch and E. Leger.—Already noticed.

Cinchonine.—E. Jungfleisch and E. Leger.—Already noticed.

Researches on the Mechanism of Etherification in Plants.—E. Charabot and A. Hébert.—Already noticed.

Some Essences of Thyme.—P. Jeancard and C. Satie.—Essences of thyme are estimated according to their proportion of phenols; it is generally considered that a pure essence should contain 25 to 30 per cent. The author, however, has examined pure essences in which the phenols varied from 5 to 60 per cent, and he has endeavoured to find the cause of these variations. To effect this he distilled the thyme with essence of ajowan, and gives a number of tabulated results. His principal conclusion is that the phenolic portions pass over at the end, and the results depend on the manner in which the distillation is carried out.

MISCELLANEOUS.

Royal Institution.—On Tuesday next, April 29th, at three o'clock, Professor F. York Powell will begin a course of three lectures at the Royal Institution on "English Kings and Kingship"; on Thursday, May 1st, Mr. A. Smith Woodward will deliver the first of three lectures on "Recent Geological Discoveries"; and on Saturday, May 3rd, Professor Walter Raleigh commences a course of three lectures on "Poets and Poetry." The Friday Evening Discourse on May 2nd will be delivered by A. E. Tutton, Esq., on "Experimental Researches on the Constitution of Crystals"; and on May 9th by Professor J. Norman Collie, his subject being "Exploration and Climbing in the Canadian Rocky Mountains."

The New Volumes of the "Encyclopædia Britannica."—We understand that within a few weeks the first of the new volumes of the "Encyclopædia Britannica" will be published jointly by Messrs. A. and C. Black and *The Times*. Soon after *The Times* issued its reprint of the ninth edition, the project of publishing additional volumes was announced, and we are informed that this plan has been carried out in such a manner that the new publication will not only complete and bring up-to-date the ninth edition, thus forming in combination with the existing volumes the tenth edition of the "Encyclopædia Britannica," but will also constitute a distinctive and independent library of reference dealing with recent history and recent progress in all departments of knowledge. The editors-in-chief are Sir Donald Mackenzie Wallace, President Hadley, of Yale University,

and Mr. Hugh Chisholm. The contents will include more than 10,000 articles by more than 1000 contributors, 150 full-page plates, 125 coloured maps, and 2300 other illustrations, eleven volumes in all. The volumes will be published at, approximately, monthly intervals. We notice, with much surprise, that though departmental editors have been appointed for almost every branch of knowledge, such as Military Affairs, Theology, Mining, Botany, Astronomy, Electricity, Music, &c., there is no department for the most important science of all, Chemistry.

The Formation of Nitride of Magnesium by the Calcination of Magnesium in the Air.—W. Eidmann and L. Moeser.—M. Mallet has already announced (CHEM. NEWS, vol. xxxviii., p. 39) the formation of a considerable quantity of nitride during the incomplete combustion of magnesium in the air. The author has examined the conditions which gave the best results, the influence of foreign substances added to the powdered magnesium, that of temperature, and the access of air rendered more or less easy. If we gradually heat over a Bunsen flame, or better still, before the blowpipe, an iron crucible filled with magnesium filings, properly covered over, but having a very small hole pierced in the cover, we obtain, after heating for fifteen to thirty minutes, a greenish-yellow mass of Mg_3N_2, up to 80 per cent of the amount of metal employed, covered with a grey layer of magnesium of variable thickness, itself covered by a layer of magnesia. —*Berichte*, vol. xxxiv., p. 390.

New Method for Determining the Molecular Weight of Ozone.—A. Ladenburg.—A bulb fitted with two taps is weighed, first full of oxygen as pure as possible, then full of ozonised oxygen, prepared with the same oxygen, and at about 8 per cent of ozone. All these operations must be carried out in a chamber in which the temperature does not vary more than a few tenths of a degree, and where the pressure remains constant. Let π be the difference between the two weights. The taps are opened in a vessel containing turpentine which absorbs the ozone; it is then weighed again, and, finally, the bulk is filled with the essence; these two last weights give the volume v of the ozone absorbed. Let d_1 and d be the specific gravities of pure ozone and oxygen, with regard to water, we get evidently $\pi = vd_1 - vd = v(d_1 - d)$. If ozone is $o\alpha$, oxygen being o_2, we have $\frac{d_1}{d} = \frac{\alpha}{2}$, and, consequently, $\pi = vd\left(\frac{\alpha}{2} - 1\right)$, from which we get α. By this means, in five experiments, we found the values of α comprised between 2·83 and 3·14, and for the molecular weight of ozone the figures comprised between 45·3 and 50·4, or a mean of 47·78.—*Berichte*, vol. xxxiv., p. 631.

NOTES AND QUERIES.

*** Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Training and Prospects of Analytical Chemists.—As a chemistry teacher, I am sometimes asked by parents for information concerning the necessary training and prospects for lads desirous of becoming analytical chemists. Many of these are in a position to enable their sons to pursue their education with little or no salary until the age of twenty-five. Will you kindly give me the following information:—

(*a*) What would be the best or adequate course of instruction, with names of suitable institutions for pursuing such courses.
(*b*) The approximate cost of such instruction, excluding scholarships.
(*c*) The average salary for capable young men at the end of their course, in the following branches:—
(1) General analytical chemists.
(2) Works chemists (dyes, &c.).
(3) Assayers.

Books giving information on above would be of advantage to me.—H. T., B.Sc.

MEETINGS FOR THE WEEK.

MONDAY, 28th.—Society of Arts, 8. (Cantor Lectures). "Glass for Optical Instruments," by Richard T. Glazebrook, M.A., D.Sc., F.R.S.

TUESDAY, 29th.—Royal Institution, 3. "English Kings and Kingship," by Prof. F. York Powell, M.A., LL.D.

WEDNESDAY, 30th.—Chemical, 5.30. Ballot for the election of Fellows. "Preparation of Absolute Alcohol from Strong Spirit," "Vapour-pressures and Boiling-point of Mixed Liquids," and "Correction of the Boiling-points of Liquid from Observed to Normal Pressure," by S. Young. "Properties of Mixtures of the Lower Alcohols," "Properties of Mixtures of the Lower Alcohols with Benzene and with Benzene and Water," "Fractional Distillation as a Method of Quantitative Analysis," and "Vapour-pressures and Specific Volumes of Isopropyl Isobutyrate," by S. Young and E. C. Fortey. "Nitrogen Bromides containing the Propionyl Group," by F. D. Chattaway.
—— Society of Arts, 8. "The Timber Resources of the Australian Commonwealth," by Edward T. Scammell.

THURSDAY, May 1st.—Royal Institution, 3. "Recent Geological Discoveries," by A. Smith Woodward, LL.D., F.R.S.
—— Royal Institution, 5. Annual Meeting.

FRIDAY, 2nd.—Royal Institution, 9. "Experimental Researches on the Constitution of Crystals," by A. E. Tutton, B.Sc., F.R.S.

SATURDAY, 3rd.—Royal Institution, 3. "Poets and Poetry," by Prof. Walter Raleigh, M.A.

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The Examinations are open only to such candidates as have complied with the Regulations.*

The Intermediate Examination will occupy at least four days, commencing on TUESDAY, JULY 22.

Examinations in the following branches of the Final Examination for the Associateship (A.I.C.) will extend over at least four days, and may commence on either JULY 15 or 22.

(*a*) Mineral Chemistry, (*b*) Metallurgical Chemistry, (*c*) Physical Chemistry, (*d*) Organic Chemistry, and (*e*) the Analysis of Food and Drugs, and of Water, including an Examination in Therapeutics, Pharmacology, and Microscopy.

The Final Examination in the branch of Biological Chemistry will be held in OCTOBER.

Applications for admission to the July Examinations should be forwarded to the Registrar not later than TUESDAY, JUNE 10, 1902, on which day the list of candidates will be closed.

(By Order of the Council)
RICHARD B. PILCHER, Registrar and Secretary.
30, Bloomsbury Square, London, W.C., April, 1902.

* "The Regulations for the Admission of Students, Associates, and Fellows of the Institute of Chemistry" may be obtained from Messrs. Blundell, Taylor, and Co., 173, Upper Thames Street, E.C., price One Shilling.

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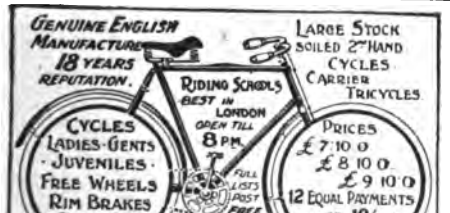
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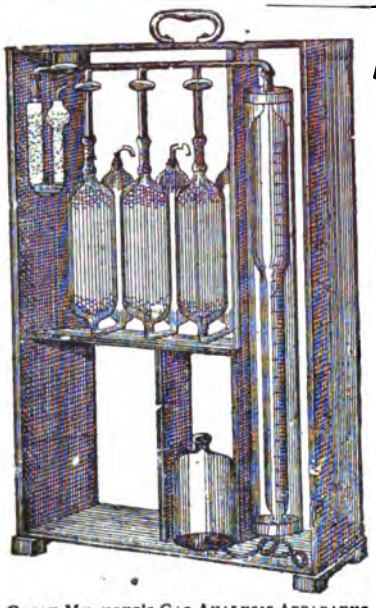

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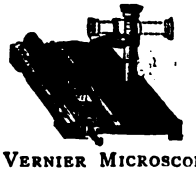

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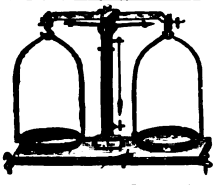

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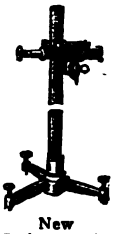

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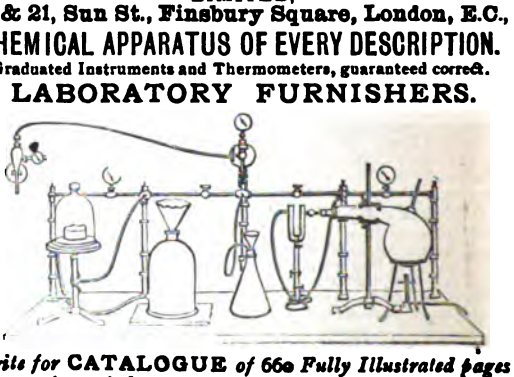

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THE CHEMICAL NEWS.

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THE RADIO-ACTIVE ELEMENTS CONSIDERED AS EXAMPLES OF ELEMENTS UNDERGOING DECOMPOSITION AT ORDINARY TEMPERATURES. TOGETHER WITH A DISCUSSION OF THEIR RELATIONSHIP TO THE OTHER ELEMENTS.

Preliminary Note.

By GEOFFREY MARTIN, B.Sc.(Lond.).

For many years there has been a general disposition to revive the ancient notion that all matter is composed of a common "protyle," and that the elements have been formed from it by a successive series of condensations.

And undoubted powerful experimental evidence has been furnished by the spectroscopic researches of Sir N. Lockyer, who believes the terrestrial elements are more or less completely dissociated into substances of a simple constitution at the high temperatures prevailing in the sun and stars. But what has hitherto been wanting is some experimental evidence that the elements do actually dissociate at temperatures attainable in the laboratory.

But it appears we have now this evidence, for the radio-active elements appear to be actually decomposing at ordinary temperatures.

Further, in all probability, this behaviour is not peculiar to the heavy radio-active elements, but occurs with every other element in the Periodic System at a suitable temperature range which differs for different elements. The only difference between the radio-active elements and the other elements of the Periodic System appears to be, that whereas in the case of the former elements this decomposition range commences at ordinary temperatures, in the case of elements of a lighter atomic weight the range occurs at higher temperatures.

In support of this view the following considerations may be urged:—A glance at Mendeleeff's periodic table will show the following gradual changes with atomic weight of the properties of the elements.

The lightest elements of any series are sharply defined and clear cut in their properties. They effect a condition of chemical repose or equilibrium in one particular state of combination; and this point of maximum stability is quite sharp and definite. In any other state of combination they are unstable. As, however, the atomic weight increases, this sharpness in combination becomes blurred, and the heavier elements of the group appear to be able to assume with ease more than one state of combination.

This shifting or blurring of the point of maximum stability of combination is always apparent with increase of atomic weight in any one group of the Periodic Table.

Now it is generally accepted that chemical combination is related to the presence of electrons in the atoms. Thus, a "ferrous" atom contains only two electrons, while a "ferric" atom contains three. If we change the iron atom from the ferrous to the ferric state we lose one electron. Interpreting the preceding relation in the light of this, it would appear that an increase in the atomic weight of the elements of any one group leads to an increased tendency to change the number of electrons in the atoms. That is to say, there is a steady decrease, with increase of atomic weight, in the stability with which the electrons are retained by the atoms of the elements. And apparently with very heavy elements—like the radio-active elements—this instability has reached such a pitch that they are even now continually casting them forth into space. So that the behaviour of the radio-active metals is only an extreme case of a tendency that exists among all the other elements of the Periodic System.

We have seen how increase of atomic weights brings about this condition of electrotonic instability among the elements. But exactly the same effect is brought about by an increase of temperature. Thus, at ordinary temperatures iron exists most stably in the ferric state. But the case is otherwise at a higher temperature. At these iron finds its state of maximum chemical repose in the ferrous state; ferric chloride and sulphate, for example, at higher temperatures decomposing to a greater or less extent into the corresponding ferrous compounds. And this behaviour is quite general among the other elements; the higher grade of combination invariably becoming more and more unstable, and the lower grade relatively more and more stable as the temperature increases.

Since every change of an elementary atom from a higher to a lower grade of combination is attended by the loss of one or more electrons, it is evident that the preceding behaviour is nothing less than the outward manifestation of the fact that, as the temperature increases, an element begins to attain a steadily increasing facility for parting with electrons usually retained by it at lower temperatures. And therefore there probably corresponds to each individual element a temperature range whereat this degree of electrotonic stability has become so marked that it spontaneously casts them forth.

When this occurs, the element is in a "radio-active" condition. So that the only difference between the so-called radio-active elements and ordinary elements is this—that ordinary temperatures are high enough to induce this condition in the heavy radio-active elements, whereas a higher temperature is necessary in the case of elements with lighter atomic weights—the temperature at which radio-activity is induced rapidly augmenting with decreasing atomic weights. According to this, radio-activity is a general condition of matter. Now, since the loss of electrons is attended with a loss of weight, it is evident that the radio-active elements are simply elements which, under given conditions of temperature and pressure, are suffering incipient decomposition, and that, further, this is only one particular example of a general decomposition which will occur among all the other elements at higher temperatures. In support of this view is the well-known fact that at high temperatures all substances become almost completely ionised. Even at ordinary temperatures it appears, from the experiments of Russell, that a great many bodies are slowly giving up electrons, and will affect a photographic plate in the dark, *e.g.*, zinc, and especially hydrogen peroxide (Chemical News, 1897, xxv., 302—396; 1898, lxxvii., 167—170).

The preceding is a mere speculation, but it is a speculation supported at every stage by experimental facts, as the following shows:—

(1). That an electric discharge shatters an atom, and reduces it to small particles, was shown experimentally by Sir W. Crookes many years ago. He called matter reduced to this fine state of division "Radiant Matter."

(2). That some of these small particles are electrons has been proved experimentally by Prof. J. J. Thomson and by Becquerel and others.

(3). That radio-active matter is at ordinary temperatures giving off electrons (and other particles?) has been proved experimentally by M. and Mdme. Curie.

(4). That hot bodies actually give off electrons has strong experimental evidence to support it.

(*a*). It is an experimental fact that gases, which at ordinary temperatures are almost absolute non-conductors of electricity, when heated to incandescence acquire a conductivity which passes that of metals at ordinary temperatures; thus arguing for the presence of free electrons.

(*b*). Carbon also greatly increases in conductivity at high temperatures.

(*c*). Hot bodies act in the same way as radio-active bodies in their power of discharging electroscopes. This was proved experimentally by Professor Worthington at the meeting of the British Association in 1888, and again quite recently by Mrs. Ayrton (*Nature*, February 27th, 1902, p. 390).

(*d*). Hot metals can charge an electroscope in the same way that radio-active bodies can. This has also been experimentally proved by Mrs. Ayrton (*loc. cit.*).

The inference, therefore, is that a high temperature causes certain bodies to give off electrons in the same way that radio-active bodies do at ordinary temperatures; *i.e.*, that heat induces radio-activity in bodies.

The proof, however, is not quite complete until it is proved experimentally that the effects observed by Mrs. Ayrton are not due to the presence of air, but will take place with as great and even greater intensity in an absolute vacuum. It is to be hoped she will see her way to make experiments on these and other points, as the matter is of great theoretical importance, and if substantiated would experimentally prove the decomposition of elements by heat into electrons (and other inactive particles ?).

(5). That heat does split up elements is shown, I believe, with the greatest precision by the experimental observations of Sir N. Lockyer on the spectra of different stars.

But what they are resolved into, whether into electrons and small particles of inactive matter as Crookes's experiments suggests, or into electrons alone, can only be decided by further experiment. In my opinion, I think it will be found that atoms when shattered give rise to three distinct kinds of particles:—

(*a*). Positively charged particles deviable in different degrees by magnetic and electrical influences.

(*b*). Negatively charged particles deviable in different degrees by magnetic and electrical influences.

(*c*). Inactive particles, composed of the unelectrified matter which composes the bulk of the atom. These particles, which may be very small indeed, would not be deviable by electric or magnetic influences.

Berlin University.
March 26, 1902.

THE MANUFACTURE OF OXIDE AND PEROXIDE OF BARIUM.

By R. HEINZ.

The increasing importance of the oxides of barium has led to many methods being devised for the improvement of their manufacture. It is more especially the production of the monoxide that has occupied attention; it enables us, in fact, to prepare hydrate of baryta or the binoxide at a low price. Any new process which solves the question in a satisfactory manner must result in great activity in this branch of industry.

The methods for the manufacture of oxide of barium start from two principles, according to the use to which the product is to be put. If we wish to produce hydrate of baryta, the decomposition of the soluble salts of barium by an alkali cannot be considered as anything beyond a laboratory experiment. If we heat carbonate of baryta in the presence of superheated steam, it frequently happens that on the addition of carbon, we do not get satisfactory results, the decomposition being incomplete. It is only by treating sulphide of barium by oxide of zinc or by other metallic oxides that, for a long time past, large quantities of caustic baryta have been prepared; this method is, however, very inconvenient, and very ill-suited to working on the large scale. On the other hand, we can obtain a very pure oxide, and, thanks to its porosity, one very well adapted to the preparation of the binoxide, by decomposing nitrate of barium at a high temperature in Hessian crucibles. The loss of nitric acid, however, makes the cost of the oxide thus prepared rather high. M. Wachtel (*Färber Zeit.*, 1900, II., 113; *Chem. Zeit. Rep.*, 1900, xxiv., 162) has given some very interesting general instructions for the manufacture of oxide and peroxide of barium.

The demand for peroxide has risen so rapidly that the resulting increase of production even cannot long remain as it is. However, as a general rule, no new factories have been started. In Italy, during the last few years, factories using the old nitrate method have been established, but with regeneration of the nitric acid; but it does not appear, up to the present, that any very advantageous results have been obtained. This method has also been tried in North America, but we have no authentic information as to the conditions of manufacture employed.

As for the peroxidation of the oxide, several new processes have been mentioned, differing slightly from those in use up to the present. These include the use of retorts arranged in various manners, of oblong or rectangular section, which can be filled and emptied easily, or muffle furnaces in several stages, so constructed that the whole furnace or each stage can be heated separately.

Thus we obtain regular heating, and the oxidation is effected regularly and easily. In practice compressed air is almost universally used for the oxidation.

With regard to the recovery of the nitric acid in the method by decomposing nitrate of barium, M. Wachtel's work already referred to may be consulted with advantage. It is claimed that 30 per cent of the total acid can be recovered (*Chem. Zeit.*, 1900, xxiv., 662), but this figure has not been verified.

As we remarked at the commencement of this article, the future has in store for us a new process of manufacture, which is a very great advance; it has been discovered at the cost of numerous and difficult experiments. The origin of this process lies in the sugar industry, more especially in the removal of sugar from molasses.

The deposits of strontianite and celestine are nearly exhausted, and new ones have not been discovered; on the other hand, it has been observed that the use of baryta completely removes the sugar from molasses, and that the product obtained is as good as that prepared by means of strontianite.

We must then, sooner or later, have recourse to baryta, and the present consumption of baryta in the sugar industry is fairly considerable. Efforts must therefore be made to establish a method for the manufacture of the oxide and hydrate of baryta which will satisfy the increasing demands which will arise. To begin with, we can only deal with carbonate of baryta, a product which can be obtained commercially in a state of great purity. Numerous attempts have been made to decompose this substance into its constituents, but all these processes are imperfect; either they are too expensive, or the decomposition is incomplete. M. Wachtel has already described several of these methods, so I shall only deal with those which he has passed over in silence.

M. Schulze proposes decomposing, by means of the electric arc, the residues from the treatment of the molasses, whether carbonates of barium or strontium. These products are mixed with carbon in the moist condition in which they are obtained, and the mass is well-stirred during the fusion; there can be no doubt that a transformation takes place, and Schulze, in small experiments, obtained 90 per cent of the oxide of barium treated, but this was perhaps rather carbide of barium, if we take into consideration the quantity of acetylene gas given off on its solution in water. According to the inventor the cost would be very low, but from analogy we might form a very different opinion; to prepare 100 kilos. of oxide of barium, about 20 kilowatt-days would be required; this represents 20 marks, reckoning the kilowatt hour at 4 pfennigs, and then we are allowing nothing for

the wear and tear of the electrodes, nor the evaporation of the water present in the residues. Further, the apparatus has a stirrer which is supposed to be used at the temperature of the electric arc, about 3000°.

Naturally, the carbonic acid formed is recovered; as for the oxide of barium, it forms very compact and hard greenish-blue masses, which the inventor of the process regards as necessary for the preparation of the binoxide. The Schenk-Bradley and Borrows-Jacobs method is approximately of the same value as the preceding one; by fusing, in the electric furnace, a mixture of heavy spar and carbon we ought to obtain a mixture of oxide and sulphide of barium. The separation of the sulphide must follow on this costly operation. To these two methods we may add that of Limb, according to which carbide of barium is prepared by fusing a mixture of sulphide of barium and carbon with a metal or a metallic oxide in the electric furnace. The carbide is decomposed with boiling water; this gives baryta, acetylene, and a residue of a metallic sulphide. All these "electric" methods seem to be based on the idea that everything can be done by electricity. The losses of original material caused by the high temperature of the electric furnace are sufficient to diminish the returns by these methods very considerably. To this must be added the intense action of oxide of barium in fusion on the sides of the apparatus, and the extremely inconvenient carburised impurities which are always to be found.

We have still to mention the practical value of a French process which consists of decomposing chloride of barium by means of oxide of zinc at a white-red heat. Chloride of zinc should be formed, which is volatilised and re-used in the form of oxide, and porous oxide of barium. Leroy and Segay's process deserves more serious attention. Carbonate of barium is decomposed at 360° by sulphuretted hydrogen, and the dry sulphide formed is treated, below a red heat, with a current of steam; in this manner oxide of barium and sulphuretted hydrogen are formed. It still remains to be seen whether this method can be worked in a practical manner on the large scale.

There is only one recent process for the decomposition of carbonate of baryta which has been proved by practical results. It can be easily worked on the large scale, and is cheap. It has been the cause of numerous interesting observations, and has caused the discovery of such interesting details that the trouble and difficulty of its invention has been to a great extent recompensed. It is an interesting fact that witherite, the natural carbonate of baryta, gives up its carbonic acid much more readily than does the artificial carbonate; therefore it is employed in certain German manufactories in spite of its slightly higher price. The installation of the process only requires comparatively simple apparatus. Two conditions are of especially great importance, viz., the construction of the furnaces and the regulation of the calcination.

The lining of the furnace with basic refractory material should be done with the greatest care if we wish the furnace to last; ordinary magnesic bricks are not at all suitable for this purpose on account of their feeble resistance. The furnace should be heated by means of gas generators; this method being the only one enabling the necessary variations of temperature to be made, which are essential to the success of the calcination. It is of great importance to be able to vary the temperature rapidly, then to increase it progressively. If the temperature is raised too rapidly, or becomes too high, volatilisation, and consequently diminution of yield, takes place. By using 100 kilos. of carbonate and 125 kilos. of fuel, we obtain, on an average, 70 kilos. of oxide of barium.

In continuous working the calcination lasts about twelve hours, and no crucibles or similar apparatus are required. It is premature to give details of this process at present; it is simple in principle, but requires incessant watching. It is important to mention that by this method oxide of barium at 95 per cent can be obtained. The product obtained is particularly suited for the preparation of a binoxide containing 90 per cent of BaO_2 free from cyanides and nitrites. The oxidation of this oxide is so easy that the peroxide can be obtained by the direct oxidation of carbonate of barium. The conduct of this operation on the large scale, however, offers certain mechanical difficulties; but the great advantage of this process is the joint production of the oxide and the binoxide of barium.

In the Hanover-Linden district a new installation has been erected at great expense for the manufacture of binoxide of barium from the oxide of barium made by the Bonnet, Ramel, Savigny, Giraud, and Marnas process. This method consists of calcining a mixture of carbon and carbonate of barium in Hessian crucible lined with paper, or some substance that will form carbon, and carrying it to 1100° or 1200°. This oxide does not seem very suitable for the production of the binoxide; according to our experiments it is a greenish-grey mass of bad appearance, and does not contain a sufficient proportion of oxygen. Our results were confirmed by the fact that this method has been abandoned after long and costly trials. The authors of this process themselves did not consider it suitable for the preparation of the binoxide, since it only gave a product suitable for this purpose after dehydration and calcining with carbon and hydrate of baryta.

The state of this industry as a whole is flourishing, and the consumption goes on increasing. At the present time France produces and exports a large quantity of hydrate of baryta, but Germany also supplies considerable quantities. On the other hand, this latter country is the principal producer of binoxide of barium. England is a serious competitor with the German manufacturers of the binoxide; other countries are of no importance in this respect, and obtain their supplies from England and Germany. The present total annual production of binoxide of barium may be put at 10,000 tons. It is especially by its production of peroxide of hydrogen that it finds many uses as a bleaching and oxidising agent.—*Chemiker Zeitung*, 1901, p. 199.

A NEW INVESTIGATION CONCERNING THE ATOMIC WEIGHT OF URANIUM.*

By THEODORE WILLIAM RICHARDS
and
BENJAMIN SHORES MERIGOLD.

(Continued from p. 201).

Preparation and Collection of Pure Uranous Bromide.

The mixture of urano-uranic oxide and carbon was placed in a porcelain boat within the larger of two "telescoping" porcelain tubes. The portion of the tube containing the oxide was heated in a Fletcher furnace, and after thoroughly sweeping out the apparatus with dry nitrogen, a mixture of dry nitrogen and bromine vapour passed over the oxide. The sublimed bromide collected near the inner end of the smaller porcelain tube. The very efficient and elaborate desiccating apparatus which served so well in the work on the atomic weights of cobalt and nickel, was very kindly given by Dr. Baxter for use in this investigation.† This apparatus, with slight modifications, was used for drying the nitrogen and bromide, and was connected by a ground glass joint with the porcelain combustion tube.

With this apparatus traces of air diffused through the annular joint between the porcelain tubes, forming a coating of oxide on the inner tube.‡ In the case of

* Contributions from the Chemical Laboratory of Harvard College. From the *Proceedings of the American Academy of Arts and Sciences*, xxxvii., No. 14.

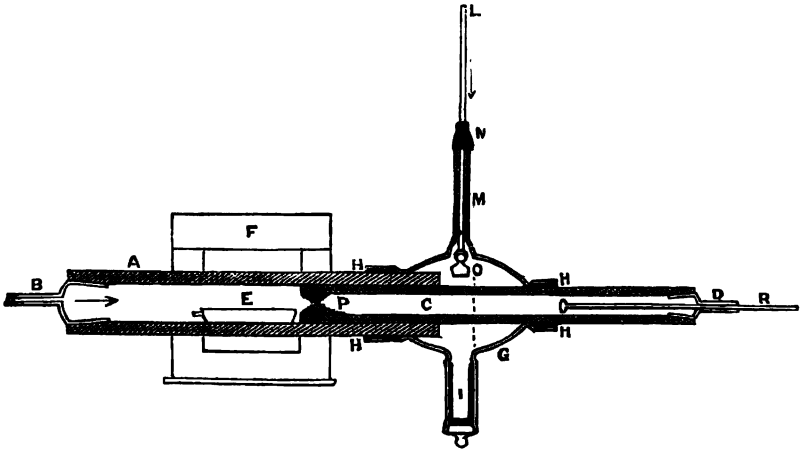
† For a full description of this apparatus see *Proc. Amer. Acad. Arts. Sci.*, 1897, xxxiii., 124.

‡ In the case of cobalt and nickel this oxide was easily removed by subsequent treatment, but in the present case removal was impossible

uranium, the oxide is found to be copiously mixed with the sublimate also. This diffusion of air takes place even when the outer end of the inner porcelain tube is nearly closed, thus making a considerable outward current within the tubes.

In order to obviate the difficulty and exclude air a glass jacket was slipped over the joint between the tubes. The construction and use of this jacket will be made clear by reference to the accompanying drawing.

The jacket was drawn down at the ends, so as to fit the porcelain tubes A and C as well as possible, and the spaces between the tubes and the jacket were packed tightly with asbestos-wool. This packing makes a joint sufficiently tight to withstand a pressure equal to that of 8 or 10 c.m. of water. The jacket was provided with a long tube, M, within which slid a second tube, L, connection being made by means of the short piece of rubber tubing, N. To the end of the inner tube was attached, by platinum wires, the stopper, O, of the weighing bottle. The outside diameter of L was very little less than the inside diameter of M, thus leaving very little space between the walls of the two tubes. For this reason, and also on account of the length of the tube M—about 15 c.m.—there was little danger of bromine diffusing up in sufficient quantities to attack the rubber connection, N. Even if this were the case there could be no possibility of contamination of the heating in nitrogen, bromine vapour was passed in through B. During the first trials of the apparatus it was our practice to keep a slow current of nitrogen passing in at L during the sublimation. This kept the jacket entirely free of bromine, a very slow current of nitrogen being sufficient to keep any bromine from passing between the walls of the porcelain tubes. It was found, however, that traces of air diffused through the permeable asbestos packing, and were of course carried into the combustion tube by the current of nitrogen, forming on the inner tube a coating of oxide, and contaminating the sublimate. In order to avoid this, the nitrogen was shut off from L some time before turning on the bromine. After turning on the bromine, the jacket slowly filled with dilute bromine vapour. While the greater part of the sublimate collected within the inner tube, a little collected between the walls of the two tubes, almost sealing the annular space. This sublimate, which collected on the outside of the inner tube, is a valuable indicator of the condition of the sublimate within. In the presence of mere traces of oxygen the lustrous brown colour of the uranous bromide gives place to a dull yellow colour easily distinguishable. Comparatively small quantities of oxygen form a coating of black oxide. When the sublimation is conducted according to the method described, the outside of the inner tube is free from any traces of the supposed oxybromide or of



SECTION OF SUBLIMING AND BOTTLING APPARATUS.

A, Outer porcelain tube fitted with ground-glass joint, B; C, inner porcelain tube with ground-glass stopper, D; E, boat containing oxide and carbon; F, furnace; G, glass jacket; H, H, H, H, packing of asbestos-wool; I, weighing bottle; L, tube for admitting nitrogen, sliding within tube M through rubber connection N, and carrying at its end stopper O of weighing bottle; P, sublimate; R, rod for removing sublimate.

sublimate thereby, since there was always a constant outward pressure of bromine during the sublimation. The outer end of L was connected with the nitrogen supply of the desiccating apparatus. All glass joints and stop-cocks were lubricated with syrupy phosphoric acid.

The method of procedure was as follows:—In the porcelain boat, E, was placed an intimate mixture of urano-uranic oxide and pure carbon, the carbon being about 20 per cent of the weight of the mixture, thus insuring a large excess of carbon. The apparatus was then thoroughly swept out by nitrogen, which enters at B and L simultaneously. After the air was completely expelled, the combustion tube was gradually raised to a high temperature by the blast lamp. Heating in a current of nitrogen was then continued for three hours at least, sometimes longer, in order to insure complete removal of all traces of air and moisture. During this and subsequent operations, the outlet of the stopper, D, of the inner tube was nearly closed by asbestos-wool, thus maintaining a constant and considerable pressure within the apparatus, and hindering the diffusion of air. After this preliminary oxide, thus showing that no appreciable quantity of moist air could have reached the innermost portions of the sublimate. The best proof of the purity of the sublimate is of course found in the agreement of analyses of substance formed under various conditions of bromine supply.

After the bromine had been run for about one and a-half hours, the sublimate was cooled for three hours in a current of nitrogen. When the tubes were thoroughly cold, nitrogen was finally passed into the jacket through L, in order to sweep out any traces of bromine that might still remain. The inner tube, containing the sublimate, was then carefully drawn out until the inner end reached a position over the mouth of the weighing bottle, indicated in the diagram by the dotted line. This can be done without seriously disturbing the asbestos packing, a rapid current of perfectly dry nitrogen being admitted meanwhile through L. By means of the glass rod, R, the sublimate was pushed out of the tube, and dropped into the weighing bottle, I. The tube, L, carrying the stopper, was then pushed down and the stopper inserted. The stopper was held by the platinum wires so lightly that

after pushing it into place the tube, L, could be withdrawn, leaving the stopper inserted in the bottle.

Thus uranous bromide was sublimed, collected, and bottled up in an atmosphere of dry nitrogen ready for weighing, without once coming in contact with the air of the laboratory. That the apparatus is effective for the purpose intended, and capable of producing material of constant composition, was shown by the first rough analyses of uranous bromide, which yielded 57·41, 57·41, and 57·42 per cent bromine respectively. These analyses were made with material that had not been purified, but served to show the constancy of composition of the sublimate; for not only was the length of time occupied in the sublimation varied, but in one case the sublimate was cooled in bromine instead of in nitrogen. Of course if an appreciable amount of an oxygen compound were formed, by diffusion of air or moisture, there would almost certainly be discrepancies in the results, since it is hardly conceivable that under the varying conditions exactly the same quantities of oxy-salt should be formed each time.

Because the specific gravity of uranous bromide was unknown, the following determinations were made:—2·0328 grms. of the salt displaced on one occasion 0·3332 grm. of kerosene at 21°, and at another trial 0·3322 grm. The kerosene had been re-distilled, and only the high boiling portion was used. The density of the kerosene at 21°, referred to water at 4°, was 0·7919. Hence the specific gravity of the uranous bromide was (1) 4·830 and (2) 4·846, giving as the mean 4·838. This value was used in reducing the observed weights of bromide to the vacuum standard.

During the weighing in the final analyses, the bromide of uranium was still surrounded by an atmosphere of pure dry nitrogen in the tightly stoppered weighing bottle. Since this bottle had been full of dry air when it was first weighed, a small correction had to be applied on this account. The difference in weight between 6·70 c.c. (the interior volume of the weighing bottle) of air and the same volume of nitrogen at 20° C. is 0·000265 grm. Of this nitrogen a grm. of uranous bromide displaced $\frac{1}{4·84}$ = 0·206 c.c., or 0·24 m.grm., while the brass weights used in weighing the bromide displaced 0·145 m.grm. of air. Hence in vacuum a grm. of uranous bromide would weigh 0·265 + 0·24 − 0·145 = 0·36 m.grm. more than the observed weight, while 2 grms. would weigh 0·265 + 2(0·24−0·145) = 0·46 more than the observed weight. All the weights given in the tables are corrected in this way to the vacuum standard.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

ANNUAL GENERAL MEETING, *March 26th*, 1902.

Prof. J. EMERSON REYNOLDS, Sc.D., V.P.R.S., President, in the Chair.

DR. HENRY and Mr. RAMAGE were appointed scrutators, and a ballot was opened for the election of Officers and Council for the ensuing year.

The PRESIDENT, in beginning his Address, said that he had the agreeable duty of congratulating the Fellows on the continued and ever increasing prosperity of the Society.

The numerical strength of the Society was 2335 on March 28th, 1901. Since that date 163 Fellows had been elected, and 3 had been reinstated by the Council, making a gross total of 2501. Of these, 32 had withdrawn, 25 had been removed for non-payment of two annual subscriptions, and 21 had died.

The actual number of Fellows to date was therefore 2423, the highest number yet reached, and the number of Foreign Members was 32.

The following Fellows have died:—F. J. Beale, J. H. Beckett, Henry Bird, Sir J. H. Gilbert, F.R.S., A. Hartridge, Alexander Hay, Lawrence Hislop, David Johnson, N. Leonard, H. G. Madan, Dr. Ira Moore, Dr. G. Harris Morris, John Paul, W. Shapleigh, Louis Siebold, Prof Maxwell Simpson, F.R.S., W. T. N. Spivey, W. Terrill, John Thomson, J. L. W. Thudichum, G. F. Wilson, F.R.S.

The most important test of the prosperity of the Society was, however to be found in the record of its work. In this respect also he had a favourable report to make. Since the last Anniversary, 181 communications had been made to the Society. Abstracts of all these had appeared in the *Proceedings*, whilst 139 had already been published in the *Transactions*. He ventured to think that the quality, generally, of the work presented was as high as in any previous year, and clearly indicated the continued enthusiasm and activity of the Fellows.

The *Transactions* for 1901 contained 146 memoirs, occupying 1411 pages; and the volume of the preceding year 127 memoirs occupying 1334 pages.

The volumes for 1901 contain 3754 abstracts of papers, published mainly in Continental journals, occupying 1496 pages, arranged as follows:—

	Pages.	No. of Abstracts.
PART I.		
Organic Chemistry	784	1530
PART II.		
General and Physical Chemistry		403
Inorganic Chemistry		376
Mineralogical Chemistry		169
Physiological Chemistry		363
Chemistry of Vegetable Physiology and Agriculture		306
Analytical Chemistry		607
	712	2224
Total in Parts I. and II. ..	1496	3574

The volume for 1901 contained a Memorial Lecture giving an account of the life-work of Rammelsberg. A set of the Memorial Lectures which had appeared up to the end of 1900 was issued in September last in a separate form.

The use of the Library by the Fellows continued to show their appreciation of it. 880 books had been borrowed, as against 810 during the corresponding period of last year. A large number of these were journals issued by post to Fellows resident in the country, and the Library Committee invited special attention to this development of the Society's usefulness. The additions to the Library comprised 153 books, 441 volumes of periodicals, and 33 pamphlets, as against 95 books, 327 volumes of periodicals, and 30 pamphlets during the corresponding period of last year.

It was desirable that the Society should have in the Library a copy of, at least, every work printed in English on chemical subjects to the end of the Eighteenth Century, and he would invite the co-operation of the Fellows in making the Library complete in this direction.

In the preparation of the new Library catalogue, the printing of which had been decided upon, advantage was taken of the opportunity to construct a new and convenient Card Catalogue, which, it was believed, would materially assist readers in making use of the Library.

It was his privilege to offer, on behalf of the Society, its warm congratulations to Dr. Schunck, to Mr. Lloyd Bullock, and to Dr. Francis, who this year have reached their sixtieth anniversary of admission to the Fellowship of the Society. He had pleasure to add that Mr. Buckton,

F.R.S., Mr. F. Claudet, and Mr. Darby have reached their Jubilee, and to them he would also convey its sincere congratulations.

Last year their illustrious senior Foreign Fellow, M. Berthelot, celebrated the fiftieth anniversary of his first scientific publication, and all countries united in expressing their admiration and respect for the veteran chemist. On behalf of the Society, he, with Dr. Gladstone and Professor Ramsay, presented a congratulatory address to M. Berthelot at the imposing function which was held in the Sorbonne on November 24th, 1901. This address had already been printed in the *Proceedings*.

During the year, the Society had joined in the celebration of the 450th anniversary of Glasgow University, and in the jubilee of Owens College, Manchester.

Considering the large number of Fellows now in the Society, the mortality is small; nevertheless, this year they had to regret the loss of 21 Fellows. This melancholy list includes the name of Sir Henry Gilbert, Past-President of this Society, and one ever devoted to its welfare. His immense work, carried out with Sir John Lawes, laid the scientific foundation of British Agriculture, and serves as the model on which all future researches must proceed. The Society was fully represented on the sad occasion when Sir Henry Gilbert was interred, and its representatives laid a wreath on his grave, while later on the Council passed a vote of condolence with his mourning relatives. In a short time, he hoped, a full obituary notice would be published by one far more competent to undertake it than he. Dr. Maxwell Simpson is another of those passed away from amongst us, full of years, leaving memories of good work well done, especially in synthetic chemistry, and of him and of the other Fellows whose life-work has closed records will also be shortly published.

Considerable discussion has taken place within the Society on the question of altering the day and hour of the Ordinary Meetings, which was raised in the address of his predecessor, Dr. Thorpe, last year. They had had two Extraordinary Meetings on this subject, and the outcome was the experiment now in operation, of trying alternate evening and afternoon meetings until the end of the present session. Until that experiment had been fairly made, the best course, obviously, was to suspend judgment on the question.

Grants amounting to £250 had been made from the Research Fund in aid of chemical investigations.

The remainder of the address dealt with certain aspects of the Periodic Law as modified by recent discovery.

Dr. Gladstone, F.R.S., proposed a vote of thanks to the President, coupled with the request that he would allow his Address to be printed in the *Transactions*.

Dr. Thorpe, C.B., F.R.S., seconded the motion, which was carried by acclamation.

The President having returned thanks,

Prof. Tilden, F.R.S., the Treasurer, in giving an account of the balance sheet, which he laid before the Society, duly audited, said:—

The receipts had been:—By admission fees and subscriptions, £4532; by sale of Journal and advertisements, £835 11s. 2d.; and by dividends on invested capital, £476 12s. 6d. The total receipts from all sources amounted to £5884 1s. 8d. The expenses had been:—On account of the Journal, £3233 0s. 2d.; on account of the Proceedings, £20 1s. 5d.; on account of the Library Catalogue, £42 14s. 4d.; on account of the Library, £429 15s. 5d.; House expenses, £239 19s. 1d. The total expenditure being £4913 10s. 6d.

The Treasurer, in concluding, proposed a vote of thanks to the auditors, which was acknowledged by Mr. Chapman.

Prof. H. B. Dixon, F.R.S., proposed a vote of thanks to the Treasurer, Secretaries, and Council.

Dr. Hewitt seconded the motion, which was unanimously adopted.

Prof. Meldola, F.R.S., responded.

The scrutators having presented their report to the President, he declared that the following had been duly elected:—

President—J. Emerson Reynolds, Sc.D., M.D., V.P.R.S.

Vice-Presidents who have filled the office of President—Sir F. A. Abel, Bart., K.C.B., D.C.L., F.R.S.; H. E. Armstrong, Ph.D., LL.D., F.R.S.; A. Crum Brown, D.Sc., LL.D., F.R.S.; Sir W. Crookes, F.R.S.; J. Dewar, M.A., LL.D., F.R.S.; J. H. Gladstone, Ph.D., D.Sc., F.R.S.; A. G. Vernon Harcourt, M.A., D.C.L., F.R.S.; H. Müller, Ph.D., LL.D., F.R.S.; W. Odling, M.B., F.R.S.; W. H. Perkin, Ph.D., LL.D., F.R.S.; Sir H. E. Roscoe, LL.D., F.R.S.; W. J. Russell, Ph.D., F.R.S.; T. E. Thorpe, C.B., LL.D., F.R.S.; A. W. Williamson, LL.D., F.R.S.

Vice-Presidents—E. Divers, M.D., D.Sc., F.R.S.; P. F. Frankland, LL.D., F.R.S.; H. McLeod, F.R.S.; R. Meldola, F.R.S.; H. A. Miers, D.Sc., F.R.S.; T. Stevenson, M.D.

Secretaries—W. R. Dunstan, M.A., F.R.S.; A. Scott, M.A., D.Sc., F.R.S.

Foreign Secretary—W. Ramsay, LL.D., F.R.S.

Treasurer—W. A. Tilden, D.Sc., F.R.S.

Other Members of Council—H. B. Baker, M.A.; F. D. Chattaway, Ph.D., D.Sc.; F. Clowes, D.Sc.; J. J. Dobbie, M.A., D.Sc.; A. E. Dixon, M.D.; M. O. Forster, Ph.D., D.Sc.; A. Harden, M.Sc., Ph.D.; J. Lewkowitsch, Ph.D.; J. E. Marsh, M.A.; S. U. Pickering, M.A., F.R.S.; J. A. Voelcker, Ph.D.; J. Walker, D.Sc., F.R.S.

Extra Meeting, March 26th, 1902.

Prof. Emerson Reynolds, Sc.D., V.P.R.S., President, in the Chair.

This meeting was held in the Theatre of the Royal Institution, by kind permission of the Managers.

Professor J. H. van 't Hoff, F.R.S., delivered the

Raoult Memorial Lecture.

François Marie Raoult was born on May 10th, 1830, at Fournes, in the Department of Nord. When he went to Paris to pursue his studies he had neither fortune nor patronage to assist him, so had to struggle to find means to keep himself. His first paper, published in 1853, contained observations on the transport of electrolytes by the galvanic current and on electrical endosmosis. In 1853 he was appointed "Aspirant repétiteur" in the Rheims Lycée, in 1855 "Régent de Physique" in the college at St. Dié, after which he was again at Rheims, and in 1860 at Bar-le-Duc, employing his spare time in working for his degree of Licencié ès-Sciences Physiques. In 1862, he proceeded to Sens, where, amidst adverse material surroundings, he prepared his thesis on Electromotive Force, for which in 1863 he obtained in Paris the degree of Docteur ès-Sciences Physiques.

The work of Raoult may be divided into three distinct parts: the physical, the chemical, and the physico-chemical. His career as a physicist begins with the thesis above referred to, by which he was marked out as an accurate and independent investigator already in advance of his time by the conclusions he based on a careful examination of facts. In the first part of this research he measured and compared in the cells of the Daniell type the amount of heat due to the chemical action with that corresponding to the electrical work produced, and showed that they were by no means identical. The second part was devoted to the decompositions in the voltameter by the galvanic current, the thermal phenomena being especially studied. It was whilst carrying out his electro- and thermo-chemical investigations that Raoult entered the Faculté des Sciences de Grenoble in 1867 as "chargé du cours de Chimie," being promoted in 1870 to the Chair of Chemistry, which he occupied until his death last year.

From this time forward a change is visible in the direc-

tion of Raoult's work, which now became of a more purely chemical kind, the problems still being looked at from the physical point of view. These problems included the examination of the gas from a "fontaine ardente," which he showed to be merely methane, the proof of presence of copper and zinc as normal constituents of the human liver, the inverting action of light on sugar. He showed that the presence of carbon dioxide in air diminished the production of carbon dioxide in the lungs, but that this effect was counteracted by the increased pulmonary action. His discovery of a basic calcium carbonate, $CaCO_3,CaO$, which acts like plaster-of-Paris on the addition of water, formed the subject of his last purely chemical paper. After this, he definitely enters upon that line of research which he has made peculiarly his own, and in which he persevered until his death.

His first paper on freezing-points was published in 1878 and was of a purely empirical nature, pointing out the proportionality existing between the lowering of the freezing-point and that of vapour-pressure (or raising of boiling-point). Probably owing to the difficulty of determining the strength of alcoholic liquors by means of their vapour-pressures or boiling-points, he tried their freezing-points. How this was related to practical applications is shown by the table appended to the paper, the liquids beginning with cider and ending with Marsala, the freezing-points varying from $-2°$ to $-10·1°$, and corresponding fairly with the proportion of alcohol. From solutions of ethyl alcohol he passed to those of other alcohols, and thence to other organic compounds, giving his well-known table relating to twenty-nine substances. His conclusion was that the product of the depression of freezing-point of a solution containing 1 grm. of substance in 100 grms. of water multiplied by the molecular weight of the dissolved substance is a constant. Two further papers in 1882 extended the freezing-point law to solvents other than water, and he showed that each solvent has its own molecular constant which, probably by the formation of double molecules of the dissolved substance, may be reduced to half the normal value. This constant is proportional to the molecular weight of the solvent (M_1) and is on an average $\frac{K}{M_1} = 0·62$. From organic compounds he passed to the more difficult cases of salt solutions, beginning with those of acids and bases, and finding that strong monobasic acids like hydrochloric acid and strong mono-acid bases like potassium hydroxide have a molecular depression of 37, whilst the weaker ones, like organic compounds, have only 18·5. He summed up his results thus:—"These facts prove that, in opposition to what I had admitted up till now, the general law does not apply to dissolved salts; on the contrary, they tend to indicate that it is applicable to the constituent radicles of the salts, almost as if these radicles were simply mixed in the solutions."

The early work of Raoult on voltaic cells was neglected, as it was much before its time, but his work on cryoscopy appeared just in the nick of time, and was soon warmly received everywhere. In 1892 he again took up his study of freezing-points which had been interrupted in 1884, but now from the point of view of the new theory of solutions, and showed that the molecular depressions of potassium chloride and sugar, as well as of sodium chloride and alcohol, were in accordance with the hypothesis of Arrhenius. In his summary of work in this direction, Raoult admitted all the conclusions of the theory of solutions, but as a thoroughly experimental investigator he objected to build upon that large but hypothetical generalisation known as the extended Law of Avogadro.

The relations existing between lowering of freezing-point and of vapour-pressures led Raoult to his tonometric work. He found (as Wüllner had already proved in some cases) that the ratio existing between the lowering of the pressure ($f-f'$), and the pressure itself (f), namely, $\frac{f-f'}{f}$, the so-called *relative lowering* of pressure was independent of the temperature. His next step was to determine the values of the *molecular relative lowering*. He thus formulated what he found:—"One molecule of a non-volatile substance (salts excepted) dissolving in 100 molecules of any volatile liquid, diminishes the vapour pressure of that liquid by an almost constant fraction of its value, which is about 1·05 per cent." This conclusion at once brought him into connection with the theory of solutions, and Raoult himself says of it, in 1887, that "the agreement between experiment and theory is thus in all respects as complete as one can desire in such matters." In 1893, along with Recoura, he gave his most general and exact expression of the results of his vapour-pressure observations.

Raoult's constitution seems to have been a vigorous one, the only indication to the contrary being his resignation, in 1892, of an additional professorship which he had held. He ought to have retired in April, 1900, on account of his having reached the age limit for the tenure of his chair, but by special decision of the Trustees he remained in office "hors cadre," a very high honour, and a proof of his unbroken vigour at that time. In his last year, in two comprehensive papers, he summed up his views on Tonometry and on Cryoscopy; it was just in time, for, with practically no premonitory symptoms, he died on April 1st, 1901.

His character as a man may be read in his papers, in which he showed activity, patience, tenacity in pursuing an aim, as much eye for detail as for the vaster horizon, absolute independence of mind, criticising or admitting without bias views of others as well as his own, and testing both with the same calm conviction that the last word must rest with experiment.

Prof. Odling, in proposing a vote of thanks to Prof. van 't Hoff, said that he would not add a word to the appreciative, unaffected, and simple account of the work of Raoult. It was so lucid and complete that coming from any one it would have been of great interest, but in this case, coming from Prof. van 't Hoff, it gave all the greater pleasure. Prof. van 't Hoff's work in inorganic chemistry had exercised a very great influence on modern chemical doctrine for a quarter of a century; his work had excited a high degree of interest amongst English chemists, and to-night he had supplemented their acquaintance with his work by giving them the opportunity of making acquaintance with himself. The early work of Prof. van 't Hoff did not, and very properly so, win its way without some opposition, which in certain Continental schools was unduly harsh. As with his theory of the chemical atom in space, so is it with the doctrine of osmotic pressure and the philosophy of solution. He could assure Prof. van 't Hoff that in no country had these been received with greater favour than in this, in spite of opposition more vivacious than weighty. They were accepted if not as the final truth, if the truth at all, at any rate as running on all fours with the truth.

The vote of thanks was seconded by Prof. Dewar, and supported by Sir William Huggins, President of the Royal Society.

PHYSICAL SOCIETY.

Ordinary Meeting, April 25th, 1902.

Prof. S. P. Thompson, President, in the Chair.

Dr. Dawson Turner exhibited and described a Mechanical Break for Induction Coils.

The use of induction coils in the production of Röntgen rays and in wireless telegraphy has made the construction of a suitable break a matter of importance. The ordinary break is unsuitable because of the wearing away at the point of contact, and there are objections to the use of mercurial breaks. The portable mechanical break which was shown by Dr. Dawson Turner consists of two metallic

rollers with their axes parallel, and kept in contact by a spring. One of the rollers has a cam attached to its spindle and can be made to rotate by means of a small electric motor. Once in each revolution the cam separates the rollers, thus making the break, and at the same time causing the second roller, which rides loose upon its axis, to turn about one-eighth of a revolution. As soon as the cam has passed the rollers are brought into contact by the spring, and the next break occurs at a different place. The wearing is thus distributed evenly over a large surface. The break is placed in a box containing alcohol or petroleum, and works best when rotating rapidly. An objection to the arrangement is the noise it makes when working. Some experiments were then shown on the discharge of electrified bodies by ultra-violet light. A disadvantage of the electric arc when used to furnish ultra-violet light for use in medicine is that the light is accompanied by heat, so that it is necessary to shield the patient from the heat without interfering with the passage of the light. A condenser spark between iron electrodes is useful because it gives a large amount of ultra-violet radiation without much heat. Dr. Turner showed that this light is capable of discharging bodies whether positively or negatively electrified. He then showed that glass and mica are opaque to the radiation, while pure rock salt is transparent.

Mr. Wilson Noble exhibited a Mechanical Break similar to the one already shown.

A roller and a disc, with their axes parallel, are placed in contact, and made to rotate in the same direction by a motor. Longitudinal slots are cut upon the surfaces of both, and the break occurs when a slot in the roller comes opposite a slot in the disc. Since the two are moving in opposite directions at their point of contact the break is very sudden. To vary the length of the break without altering the rate of rotation, the slot in the roller is wider at one end than the other, and the disc can be placed so as to touch the roller at any point of its length.

The Chairman said he knew of no metal which would withstand continual sparking without wearing, and suggested that breaks should be so designed that the parts affected by the sparking could easily be replaced. He asked if there was any difference in the wear of the two rollers. Referring to an experiment shown by Dr. Turner in which ultra-violet light discharged a positively electrified body much quicker than a negatively electrified one, he asked if this would be the case if the apparatus were surrounded by an earthed metallic screen.

Mr. Watson suggested that the wearing of the edges in Mr. Wilson Noble's break might be lessened by filling the cavities with slate.

Mr. Duddell said that the worst substance for making contact pieces was carbon; then came zinc, brass, and copper, with platinum best. These were the results in air, but the effects in liquids were different. With rapidly moving contacts all metals are equally good, but in ordinary coils the contact pieces move slowly.

Prof. Everett said it was interesting that rock salt, which is very transparent to long waves, should also be transparent to very short waves.

Dr. Dawson Turner said that if the experiment referred to by the Chairman had been performed inside an earthed metallic screen the negatively electrified body would have been more quickly discharged.

Mr. R. S. Whipple exhibited a Temperature Indicator for Use with Platinum Thermometers, in which readings are automatically reduced to the gas scale.

The instrument is very similar to the well-known Callendar and Griffiths temperature indicator, with the exception that it is so arranged that the readings obtained are automatically reduced to the gas scale, thus avoiding the necessity of applying a correction. It consists of a simple Wheatstone's bridge with equal ratio arms, the other arms being the thermometer and a long helical bridge wire together with the compensating leads. A travelling contact is moved round the wire until a balance is obtained. The bridge wire is wound on an ebonite drum, on the outer surface of which a helix has been cut. The contact piece, which is connected electrically with the galvanometer, is carried from the inside of a cylinder fixed to a shaft. A white celluloid tube on which the scale is divided is fixed to the outer surface of the cylinder. A screw of the same pitch as the helix on the ebonite drum is cut on the shaft, so that by rotating the shaft the contact is caused to travel along the bridge wire, and at the same time the scale is carried past an index placed above it. The scale has been so constructed that the reading at the index gives directly the temperature of the thermometer reduced to the gas scale. The instrument reads from 0° to 1400° C.

Dr. Chree asked the following questions:—Was not the instrument so constructed restricted to platinum thermometers of wire having a given specified value of δ; if so, what was this value? Did the helical wire balance the entire resistance of the thermometer, or only the excess above the resistance at 0° C., and could the instrument measure temperatures below 0° C.? What error was likely to come in when measuring temperatures up to 1000° C. from changes in the resistance of the helical wire under the variations of atmospheric temperature to which it would be exposed in ordinary use? Would not there be some wear between the wire and the contact-piece?

Dr. Harker asked what were the limits of the value of δ for wires now supplied for commercial purposes. He thought that the accumulation of dust between the contact-piece and the wire, due to continual scraping, was a more serious objection than the wearing away of the wire.

Mr. Whipple said the scale was calculated for a value of $\delta = 1{\cdot}5$. The value of δ seldom exceeded 1·52, and wire could be obtained with $\delta = 1{\cdot}5$. The instrument shown was made to read from 0° C., but could be easily arranged to read below 0° C. The wearing between the contact-piece and bridge-wire was very slight.

Mr. S. A. F. White read a "*Note on the Compound Pendulum.*"

In the determination of the length of the equivalent simple pendulum for a compound pendulum whose form is a symmetrical bar and bob, with one fixed, one movable knife-edge, and no sliding weight, it is convenient to make the mass of the movable knife-edge small. In this case small displacements of this knife-edge will not materially alter the position of the centre of gravity or radius of gyration of the pendulum about an axis through its centre of gravity. The time of swing about the fixed knife-edge will therefore remain practically constant. The best determination of the correct position of the movable knife-edge for an equal time of oscillation will be given when, for the smallest displacement of this knife-edge, there is the greatest variation in the time of oscillation about it. The author has determined the position which makes $\frac{dt}{dh}$ a maximum, h being the distance of the axis of suspension from the centre of gravity.

He has also drawn the curve showing the relation between $\frac{dt}{dh}$ and h. The calculations have then been applied to the determination of the position of the movable knife-edge in a particular pendulum. The experimental value of the ratio of h to k deduced from this pendulum when the movable knife-edge is adjusted to its right position, agrees well with that predicted by the theory.

The author states that when the length of the equivalent simple pendulum is about a metre, it should be possible with a stop-watch reading to 0·2 second, to determine g to about 0·1 or 0·2 per cent. If the fixed knife-edge were made the movable knife-edge, the value of $\frac{dt}{dh}$ would be very large, but there would be difficulties in the

way of measuring the small time of swing and the small equivalent length.

Dr. Chree said that the mathematical work might apply to either of two distinct problems. It might refer to the case of an ordinary pendulum with one movable knife-edge, the object sought being the particular position in which a given change in the period answered to the greatest movement of the knife-edge. The second case, the one which he supposed the author had in view, was that of a Kater pendulum, the object being to ascertain what design enabled the position of the movable knife-edge to be most accurately determined from the condition that the period about it should equal that about the fixed knife-edge. The author's reasons for neglecting the solution answering to a short pendulum of large moment of inertia did not appear mathematically complete, though they might be supported from physical considerations as to the relative accuracy of the determinations of g with long and with short pendulums.

Mr. Watson said that, as a practical problem for students, the length was the most difficult thing to measure. The position of the movable knife-edge for strict equality of period could be deduced by interpolation from periods determined in two positions of the movable edge. In accurate work a light bob, equal in size to the heavy bob, might be placed upon the other end of the pendulum to keep the air friction constant.

The Society then adjourned until May 9th.

NOTICES OF BOOKS.

Calorimetry of Producer and Illuminating Gases. With Special Reference to Future Legislation. By John F. Simmance, Assoc.M.Inst.C.E., M.Inst.Mech.E. London: Walter King. Pp. 30.

This volume does not propose to deal in any way with the theoretical heat values of gases, but is intended to be a practical guide for the operator who desires to estimate, rapidly and accurately, their practical calorific effects in recognised units. It is obvious that for different tests to be comparable they must be carried out in a similar manner. This is done with regard to the illuminating power of coal-gas, but with gas calorimetry no such uniformity of working or of apparatus has hitherto been attempted, and it is with the object of effecting this that the present present code of instructions has been prepared.

In modern gas calorimetry the amount of heat imparted by the combustion of a given volume of gas to an ascertained quantity of water is the basis for estimating the calorific power of the gas.

The calorimeter is, briefly, a means of enveloping the flame in a shell of water which is regularly changed, and the temperatures of which before and after passing the instrument are accurately gauged, as is also the after-temperature of the products of combustion. The essential points of the test are:—

1. The accurate measurement and invariability of the gas flow.
2. The same as to the water.
3. The correct reading of the thermometers.
4. The obviation of any outside influences which might induce errors in the above.

These four principal points are considered in turn, and a number of useful hints are given to help in the satisfactory carrying out of the tests.

A detailed account of the tests then follows, in which most minute instructions are given, making it next to impossible for any one who reads this book to make a mistake. If is pointed out that the most likely cause of difference between two operators is the "personal error"; this, however, can be entirely abolished by making the gas meter automatically measure the water as well, when the operator will only have the thermometers and the water-gauge to attend to.

The Year-Book of New South Wales. Compiled by the Editor of the "Year-book of Australia," for Circulation by the Agent-General in London. 1902. Pp. 168.

This book is compiled somewhat on the lines of the well-known Whitaker's Almanack, though on a much smaller scale. It contains a large amount of political, historical, and commercial information, which bears ample evidence of the increased industrial importance of the country. So rapid has this development been that it is found necessary to have a specially appointed resident official in London for each of the Australian Colonies, these gentlemen being called the Agents-General. They are intrusted with all external business, especially that of a financial character, but it is also a portion of their duties to supply those desiring it with any information procurable respecting their several Colonies.

In these pages we find a good account of the mineral resources of the Colony, accompanied by a large coloured map showing the districts in which the various minerals are met with. The water supply is also fully dealt with, and many interesting particulars are given. Sydney appears to have an exceptionally large supply; the main storage reservoir at Prospect, about twenty-two miles from the town, has a gross capacity of 11,392,713,000 gallons, and an available capacity of 7,324,343,000 gallons; this reservoir is formed by a dam 84·7 ft. high, 30 ft. wide on the top, and 7400 ft. long, giving a water surface of 1261 acres, or nearly two square miles.

The important progress that the mining industry of New South Wales has made is apparent when looking through the statistics given. Every decade from 1831-40 to 1891-1900 shows a large and growing increase, the total value to the end of 1900 being £140,635,533, among which we notice noble opals £456,599, and diamonds £55,535, besides the well known large returns of gold, silver, copper, tin, coal, &c.

The Chemical and Physical Examination of Portland Cement. By Richard K. Meade, B.S., Instructor in Chemistry in Lafayette College. Easton, Pa.: The Chemical Publishing Company. 1901. Pp. 183.

In preparing this manual the author has kept in view the possibility that many cement manufacturing companies are employing young men fresh from technical schools whose college training may be excellent, but whose practical experience is usually limited to a few analyses, while their knowledge of making the physical tests is in all probability even less; for this reason both the chemical and physical examination of cements have been explained very fully and with abundance of detail.

Tricalcium silicate is the essential element of Portland cement, in which it occurs in cubical crystals; in this compound the lime and silica bear the ratio of 2·78 to 1. Le Chatelier concluded from his researches that the tricalcium silicate when mixed with water reacts to form a hydrated monocalcium silicate and calcium hydroxide, according to the reaction:—

$$2Ca_3SiO_5+9H_2O=(CaSiO_3)_2.5H_2O+4Ca(OH)_2.$$

The calcium hydroxide then probably reacts further upon the calcium aluminate of the cement, forming hydrated, basic, calcium aluminate, $Ca_4Al_2O_7.12H_2O$. The "hardening" of cements is due to the first reaction, but the formation of the hydrated basic calcium aluminate probably exerts a marked influence on the "setting."

On the other hand, Messrs. S. B. and W. B. Newberry arrived at different conclusions, viz., that a correctly balanced Portland cement should be made as follows:—Per cent of lime = per cent of silica × 2·8 + per cent of alumina × 1·1. The ratio between the silica and alumina on the one hand and the lime on the other is termed the "hydraulic index."

The principal constituents to be determined in the analysis of cements are the silica, ferric oxide, alumina, lime, magnesia, and carbonic acid. Analytically, cements lie on the one side between limestones and marls, which

are easily decomposed by dilute hydrochloric acid, and on the other, clays and refractory silicates requiring fusion with alkaline carbonates for their decomposition. Different methods for all these estimations are given, both gravimetric and volumetric, besides rapid methods for the analysis of cement mixtures, slurry, &c.

The value of a cement depends on its physical properties, more especially its setting power, tensile strength, and soundness; a quick-setting cement, one setting in less than half-an-hour, is preferable for submarine work, while for most purposes, where sufficient time will be given the cement to harden before being brought into use, a slow-setting cement usually answers better.

While in actual work cement is never used in such a way as to subject it to tensile stress, still the test of the tensile strength is the most convenient, as the ratio between the tensile and compressive strength of a cement has been found to be a fairly constant one, various methods of applying this test are fully described. The book, which is well adapted to be of practical use, closes with a number of reference tables and an index.

A College Text-book of Chemistry. By Ira Remsen. London: Macmillan and Co., Ltd. 1901.

This book is intended to be intermediate in difficulty between the "Introduction to the Study of Chemistry" and the "Inorganic Chemistry" of this series. According to the old familiar method, the several elements, grouped according to Lothar Meyer's system of natural families, are studied successively in detail; thus there is nothing new in the arrangement of the book; on the other hand, it is probably hardly necessary to say that the text is accurate and thorough. The subject-matter of every chapter is illustrated by a few experiments, admirably described in all cases with a sufficiency, but not a superfluity, of detail; the suggestive questions addressed to the student should fulfil their purpose of making him think of, and reason about, what he is doing; and we feel inclined to believe that if they fail in exciting and keeping alive his interest he will profit but little by the study of natural science. The first half of the book is devoted to the principal elements of families VII. to IV., and the compounds they form with one another. Chapters XXIII. and XXIV., which deal with the theory of chemical action and general considerations relating to the base-forming elements, admirably demonstrate the author's capabilities in the direction of lucid explanations and far-reaching generalisations. The rest of the book is occupied with the metallic elements and familiar compounds of carbon.

Beiträge zur Chemischen Physiologie und Pathologie. Edited by Franz Hofmeister. Band II., 1-3 Heft. Braunschweig: Friedrich Viewig und Sohn. 1902.

The first paper of the March number of this journal deals with the effects of the addition of various substances upon the solidifying and melting-points of gelatin. The substances added were, firstly, single inorganic salts; secondly, two salts, either with or without common ions; thirdly, a mixture of an electrolyte and a non-electrolyte; and lastly, non-electrolytes. The results are graphically represented in fourteen curves. This paper is the first instalment of further communications concerning similar researches on albuminous bodies. The second paper, by Dr. Hugo Wiener, on the synthetic formation of uric acid in the animal body, contains a number of tables showing the influence of the addition of various organic substances either *per os* or subcutaneously on the formation of uric acid in birds and mammals respectively. The general result appears to be that the substances employed with a chain containing three carbon atoms up to propionic acid produce an increased formation of uric acid, while all substances with a chain of four carbon atoms up to β-oxybutyric acid have no effect.

In another of the eight articles contained in this number is discussed the occurrence in the pancreas of an enzyme, similar to zymase, which is capable of splitting up dextrose and forming carbon dioxide and alcohol. The question presents considerable difficulties when attacked experimentally, and the researches described appear to leave the matter still undecided.

Thermodynamique et Chimie. By P. Duhem. Paris: Librairie Scientifique, A. Hermann. 1902.

In the first five chapters of this work, which is intended for the use of chemists, the fundamental principles underlying chemical statics and dynamics are thoroughly examined and formulated, and an attempt is made to exclude as far as possible all references to higher algebra, so that the student who does not possess any considerable knowledge of mathematics will be able to follow the author's reasoning without difficulty. A special feature of the book is the great amount of attention paid to recent developments and applications of thermodynamics. If in places we feel that the importance of some of these applications is exaggerated, and that some of the views put forward are unduly biassed, we are, nevertheless, conscious that Professor Duhem has conferred a real benefit on chemists generally by bringing his great knowledge of physics to bear upon the question of the simplification of the difficult subject of themodynamics.

CORRESPONDENCE.

ARSENITE OF SILVER.

To the Editor of the Chemical News.

Sir,—Your correspondent "Rip van Winkle" asserts, in effect, that the composition of the canary-yellow arsenite of silver was finally settled by Bloxam's paper published forty years ago in the *Journal of the Chemical Society*. But careful examination of the literature of that period shows that Bloxam's paper, so far from clearing up the mystery of the composition of the canary-yellow, left that question in greater confusion than ever.

Before Bloxam's publication the canary-yellow was supposed to be one definite chemical substance, or, at any rate, to be of approximately uniform composition. Bloxam's paper imports a doubt.

There is only one complete analysis of any specimen of the canary-yellow, and the analytical figures lie about midway between the Pasteur formula and the Filhol formula. The figures are:—

AgO	74·77
As_2O_3	25·88
	100·65

Bloxam's surmise as to the nature of that specimen of canary-yellow was that it consisted of Filhol's arsenite mixed with a quantity of arsenious acid.

To the filtrate from that specimen of the canary-yellow more solution of nitrate of silver was added, and also ammonia, and a further precipitation of canary-yellow took place. That second specimen of canary-yellow was not fully analysed, the only analytical figure being 22·19 per cent of As_2O_3. That figure accords with the Filhol formula, and would have been important if it had been associated with a concordant silver determination.

In the absence of the necessary silver determination it establishes nothing at all; inasmuch as the drop in percentage of As_2O_3 from 25·88 to 22·19 might have been due to deposit of saltpetre or nitrate of silver in the precipitate. In short, there was surmise when there might have been certainty; and Pasteur's erroneous composition of the canary-yellow arsenite was not rectified in the year 1862.

Under these circumstances we need not wonder that the Pasteur formula for the canary-yellow found its way into "Watts's Dictionary" in the year 1863, when the first volume was issued, and that it remained in possession of the field until the completion of the Dictionary in the year 1868. Neither need we wonder that so well-informed and so careful a chemist as Dittmar retained the Pasteur formula in one of his books issued in the year 1876.

In conclusion, I have to state that "Rip van Winkle's" assertion that my recently published work was anticipated by Bloxam is absolutely devoid of foundation.

In some very important aspects Bloxam's results are the very opposite of mine. Bloxam's canary-yellow was of variable composition.

In my hands the canary-yellow arsenite has exhibited the utmost constancy of composition.

In my paper there is a record of a complete analysis of the pure canary-yellow; in Bloxam's paper no such record is to be found.—I am, &c.,

J. ALFRED WANKLYN.

The Laboratory, New Malden Surrey.
April 27, 1902.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

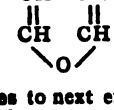
NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxiv., No. 14, April 7, 1902.

A Type of the Compounds of Beryllium.—H. Lacombe.—The author succeeds in preparing homologues superior to beryllium acetate, $(C_2H_3O_2)_6Be_4O$; this latter substance being prepared last year. All the compounds obtained are of the type A_6Be_4O, A representing the radicle of a fatty acid. He has not yet succeeded in preparing salts of the type A_6Be_4O with strong mineral acids.

Constitution of the Chlorohydrines.—Marc Tiffeneau.—In 1875 Markovnikoff showed, contrary to Henry's assertions, that during the fixation of ClOH on to propylene, the oxyhydrile takes the least number of hydrogen atoms on to the carbon, giving chlorohydrine, $CH_3—CHOH—CH_2Cl$. However, he attributes to isobutylene chlorohydrine the formula $\frac{CH_3}{CH_3}>CCl—CH_2OH$, apparently showing that it is not possible to establish a general rule for the fixation of ClOH on to the ethylenic carbides. The author now verifies Markovnikoff's rule that, during the fixation of ClOH on to the ethylenic carbides, the oxyhydrile always takes the least hydrogen on to its carbon atom.

Nitration of Furfurane, and a Derivative of Nitrosuccinic Aldehyde.—M. Marquis.—During the nitration of furfurane in acetic anhydride solution, the ring is opened, with formation of a monoacetine of nitrosuccinic aldehyde. Pyridine is able to act on this substance, closing the ring by raising the elements of acetic acid, and giving a nitrofurfurane whose constitution is evidently the following:—



The author proposes to next examine the aldehydic product of the action of water on nitrosuccinic acetine.

New Method of Preparation of Oxygen.—George F. Jaubert.—The peroxides of sodium and potassium are very rich in active oxygen; that is, oxygen which can easily be evolved in the gaseous state. Sodium peroxide contains 20·5 per cent, potassium 33·8 per cent of such oxygen. The author endeavours to utilise these bodies for the industrial preparation of oxygen and facilitate its change into the latent state in a solid body susceptible of giving it up in the cold under the simple action of water. The sodium peroxide is made into blocks by simple compression after being mixed with the theoretical quantity of a soluble permanganate (Na, K, or Ca) or of a hypochlorite, the latter substance facilitating the decomposition of the hydrate of sodium peroxide which forms under the action of water and is stable in the cold. The oxygen obtained by such a method is interesting from several points of view, chiefly on account of the ease of formation, —an apparatus of the nature of Kipp's sufficing to prepare thousands of litres,—and also on account of the absolute purity of the oxygen prepared.

Classification and Atomic Weights of Neon, Argon, Krypton, and Xenon.—H. Wilde.—In a previous paper on the atomic weights of argon and helium, the author finds the position of argon amongst the elements comes near to nitrogen in the series $H \times 7n$, with an atomic weight 21. The elements recently discovered, neon, krypton, and xenon, entirely confirm the position and the atomic weight of argon in this table; and the most recent determinations of the densities of these new gases by Ramsay and Travers show that they belong to the series $H \times 7n$. The gaseous members of the series $H \times 7n$ are arranged in the following order:—Ne, 7; N, 14; Ar, 21; Kr, 42; Xe, 63.

MISCELLANEOUS.

Iron and Steel Institute.—The Annual Meeting of the Institute will be held at the Institution of Civil Engineers, Great George Street, Westminster, on Wednesday and Thursday, the 7th and 8th of May, 1902, commencing each day at 10.30 a.m.

Programme of Proceedings.

Wednesday, May 7th, at 10.30 a.m.—General Meeting of Members. The Council will present their Report for the year 1901. The Hon. Treasurer will present the Statement of Account for 1901. Scrutineers will be appointed for the examination of the Voting Papers. Election of Officers and Council. The Bessemer Gold Medal for 1902 will be presented to His Excellency F. A. Krupp, of Essen. A selection of Papers will be read and discussed.

At 7 p.m., Annual Dinner of the Institute in the Grand Hall of the Hotel Cecil.

Thursday, May 8th, at 10.30 a.m.—General Meeting of the Members at the Institution of Civil Engineers. The following is a list of the Papers that are expected to be submitted:—

Report by the Committee appointed to investigate the Nomenclature of Metallography.

"On a New Vacuum Tuyere for Blast Furnaces." By Horace Allen, London.

"On the Microstructure of Hardened Steel." By Prof. J. O. Arnold and A. McWilliam, Sheffield.

"On the Compression of Fuel before Coking." By J. H. Darby, Brymbo.

"On Gas from Wood for Use in the Manufacture of Steel." By James Douglas, LL.D., New York.

"On a Combined Blast-furnace and Open-hearth Process." By P. Eyermann, Benrath, near Düsseldorf.

"On the Physical and Chemical Properties of Carbon in the Hearth of the Blast Furnace." By W. J. Foster, Darlaston.

"On the Sulphur Contents of Slags and other Metallurgical Products." By Baron H. von Jüptner, Donawitz, Austria.

"On the Elimination of Silicon in the Acid Open-hearth Furnace." By A. McWilliam, Sheffield, and W. H. Hatfield, Sheffield.

"Report on Research Work carried out during the Past Year." By J. A. Mathews, Ph.D., New York (Andrew Carnegie Research Scholar).

"On the Iron Ore of Brazil." By H. Kilburn Scott, Rio de Janeiro.

"On the Recovery of By-products in Coking." By J. Thiry, London.

"On Brinell's Researches on the Influence of Chemical Composition on the Soundness of Steel Ingots." By Axel Wahlberg, Stockholm.

The Autumn Meeting of the Institute will be held in Düsseldorf on September 2nd and following days.

"Mechanical Refrigeration."—Messrs. Whittaker and Co. announce that they have in the press and will shortly publish in their Specialist Series a work entitled "Mechanical Refrigeration." The volume, which is by Mr. Hal Williams, A.M.I.Mech.E., will deal with the whole field of ice making and cold-storage, and while designed for the use of those technically interested in the industry, and is in all respects a practical book, is an attempt to express in simple language the principles regulating the action of refrigerating machinery, and the latest and most up-to-date practice in its application.

MEETINGS FOR THE WEEK.

MONDAY, 5th.—Society of Arts, 8. (Cantor Lectures). "Glass for Optical Instruments," by Richard T. Glazebrook, M.A., D.Sc., F.R.S.
—— Royal Institution, 5. General Monthly Meeting.
—— Society of Chemical Industry, 8. "On the Mixed Carbides of Manganese and Calcium," by J. S. Brame and Prof. Vivian B. Lewes. "Dangerous Chemical Substances," by Oscar Guttmann, F.I.C.

TUESDAY, 6th.—Royal Institution, 3. "English Kings and Kingship," by Prof. F. York Powell, M.A., LL.D.
—— Society of Arts, 8. "The Printing of Modern Illustrated or Decorated Books," by Charles T. Jacobi.

WEDNESDAY, 7th.—Society of Arts, 8. "Origin and History of Carriages," by Albert Chancellor, J.P.
—— Iron and Steel Institute. Annual Meeting, 10.30 a.m. Dinner, 7 p.m.

THURSDAY, 8th.—Iron and Steel Institute. General Meeting, 10.30 a.m.
—— Royal Institution, 3. "Recent Geological Discoveries," by A. Smith Woodward, LL.D., F.R.S.
—— Society of Arts, 4.30. "Past and Present connection of England with the Persian Gulf," by Thomas Jewell Bennett.

FRIDAY, 9th.—Royal Institution, 9. "Exploration and Climbing in the Canadian Rocky Mountains," by Prof. J. Norman Collie, Ph.D., F.R.S.

SATURDAY, 10th.—Royal Institution, 3. "Poets and Poetry," by Prof. Walter Raleigh, M.A.

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Botany..	O. B. Rendle, M.A., D.Sc.
Zoology	H. W. Unthank, B.A., B.Sc.
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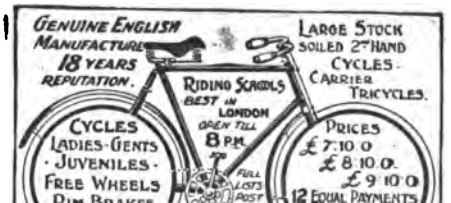
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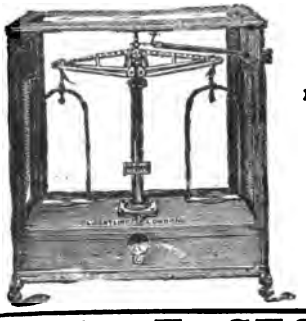

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THE CHEMICAL NEWS

Vol. LXXXV., No. 2215.

FURTHER NOTE ON MINERALS OCCURRING IN AUSTRALIAN BAT GUANO.

By R. W. EMERSON MacIVOR, F.I.C., &c.

Mr. L. Fletcher, F.R.S., the distinguished head of the Mineralogical Department of the British Museum, writes me a courteous letter, in which he points out that the name Müllerite which I proposed to give to the new ammonium-magnesium phosphate, $Mg(NH_4)_2H_2(PO_4)_2+4H_2O$, described in my recent paper in the Chemical News (vol. lxxxv., p. 182), has been used lately by Zambonini (*Zeit. Kryst. Min.*, 1899, vol. xxxii., p. 157) for a mineral having the composition $Fe_2O_33SiO_2.2H_2O$; and also that it had previously been used for a "Krennerite-like mineral."

"I am afraid," says Mr. Fletcher, "that Zambonini's description has the priority as regards this assignation to a species with determined character (specific). The confusion is regrettable, and perhaps the best and simplest plan would be for you to give a new name in substitution for the one given so long ago."

In compliance with this suggestion, and to prevent all future confusion, I have now decided to call this interesting mineral Schertalite, in honour of an old master, Prof. Arnulf Schertal, late of Freiberg, Saxony.

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AN INFRA-GASEOUS STATE OF MATTER.

By H. STANLEY.

It is now about a quarter of a century since Sir William Crookes enunciated his theory of the ultra-gaseous state of existence of matter, and what he then termed "radiant matter" now poses as "electrons."

Suppose we have a gas confined in a cylinder fitted with a piston which will enable us to keep the pressure constant as we cool down the gas. Let this pressure be less than the critical pressure and greater than zero. Then let the temperature be speedily reduced to absolute zero.

Considering this in the light of Van der Waals's well known theorem, we have—

$$\left(p + \frac{a}{v^2}\right)\left(v - b\right) = 0.$$

Since, by hypothesis, the pressure is not zero, we must have—

$$v = b.$$

Now, at absolute zero, the molecules of a gas will be dormant as far as energy of translation is concerned, but there will still remain an attractive force between the molecules, viz.,—

$$\frac{a}{b^2}.$$

This force will be proportional to the product of atomic masses or some similar quantity, independent of temperature.

According to Van der Waals, b is four times, and according to O. E. Meyer $4\sqrt{2}$ times, the space occupied by the molecules. Now the heat developed by this attractive force will be measured by—

$$\int_{b_1}^{b_2} \frac{a}{b^2} db = a\left(\frac{1}{b_1} - \frac{1}{b_2}\right);$$

where b_1 is the quantity defined as above (*i.e.*, a constant times the volume of the molecules), and b_2 is the absolute volume of the molecules.

If we define the molecule as a cluster of quasi-Boscovich atoms of non-spherical shape (see Kelvin, *Phil. Mag.*, 1901), by their mutual attraction they will agglomerate irregularly, and in so doing will yield up heat according to the above expression. If, now, the pressure is increased till we reach a critical value, the molecules will assume a regular and definite position with respect to each other (*i.e.*, condense). There is thus an intermediate or "infra-gaseous" state between the gas and liquid.

Note.—Since writing the above, I have read with interest Brinkworth and Martin's paper on a somewhat similar question ("The Heatless Condition of Matter," Chemical News, vol. lxxxv., p. 194). Is their "heat-point" the temperature at which the "infra-gaseous" state exists? as the heat developed, according to the foregoing equation, if not allowed to escape, would raise the temperature of the gas by a definite amount, which would vary with the nature of the gas.

Merchant Venturers' College,
Bristol, April, 1902.

THE ACTION OF SOME REAGENTS ON AMORPHOUS SILICON.

By P. LEBEAU.

With the object of deciding whether free silicon would be likely to be present in the products resulting from the attack of siliceous cast-iron by different compounds, we were led to examine the action of certain reagents on amorphous silicon.

Having only been able to find very scanty information on this subject, we thought it would be of interest to publish the results of our experiments.

As it seemed reasonable to admit that the free silicon existing in metallurgical products containing only a very small proportion of this metalloid would be in a very fine state of division, we made our experiments on the most finely-divided silicon we were able to prepare. For this purpose we separated, by means of levigation, the lightest portions of an amorphous silicon obtained by M. Vigouroux's method. The action of each reagent was examined in the following manner:—

A certain quantity of the thoroughly dried amorphous silicon was weighed out, and it was acted on by the liquids usually used for the attack of cast-iron, operating either in free air in a porcelain crucible, or in a conical Bohemian flask fitted with a cork and two glass tubes to allow the passage of a current of carbonic acid.

The duration of each experiment was about six hours, and the temperature was maintained at about 100°. At the end of this time the solution was poured on to a tared filter to collect the silicon, which was washed successively with hydrochloric acid at one-tenth per cent, then cold water, and finally with boiling water. The desiccation was effected in an oven at 80°. The following are the results obtained:—

Nature of the reagent.	Weight of silicon used.	Weight of silicon found	
		In free air.	In the current of CO_2.
	Grm.	Grm.	Grm.
Solution of cupric chloride at 10 per cent	0·200	0·203	0·2004
Solution of cupric sulphate at 10 per cent	0·200	0·2002	0·2012
Solution of $CuCl_2+NH_4Cl$ at 10 per cent	0 200	0·1996	0·2008
Solution of $CuCl_2+KCl$ at 10 per cent	0·200	0·2015	0 1998
Nitric acid with its own volume of H_2O	0·302	0·3015	—

Our experiment with nitric acid only confirmed the previous experiments of M. Vigouroux (*Ann. Chim. Phys.*, Series 7, vol. xii., p. 5) on the action of this liquid on silicon, which he mentioned in his paper on the properties of this metalloid.

We also used a ten per cent solution of chromic acid under the same conditions.

The silicon was weighed on a tared filter after the destruction of the chromic acid by hydrochloric acid and alcohol.

Weight of silicon used .. .. 0·2372 grm.
Weight of silicon found.. .. 0·2363 „

As in the attack of castings a ferrous or a ferric salt is produced, we thought it advisable to examine in the same way the action of a solution of ferric chloride and that of a solution of ferrous chloride.

	Weight of silicon used. Grm.	Weight of silicon found. Grm.
Ferric chloride at 10 per cent ..	0·3135	0·3133
Ferrous chloride at 10 per cent ..	0·229	0·2296

The result of all these experiments clearly shows the resistance of silicon, even when finely divided, to the reagents generally used for the attack of cast-iron and steels. We can thus be certain that its absence in the residues from these attacks arises from the fact that the silicon is present in combination and not in the free state. —*Bull. Soc. Chim.*, Series 3, vol. xxvii., No. 3.

ESTIMATION OF THE SILICON IN RICH FERRO-SILICONS BY MEANS OF PEROXIDE OF SODIUM.

By Dr. C. RAMORINO.

The use of peroxide of sodium for the determination of silicon in rich ferro-silicons (50 per cent and over) has not yet been applied practically in laboratories, on account of the difficulty found in working with this compound. The reaction which takes place if we attempt directly to decompose a ferro-silicon rich in silicon and carbon by means of the peroxide is very violent, on account of the powerful exothermic reaction which takes place. Reduction occurs suddenly, accompanied by projections and perforation of the platinum crucible. On the other hand, the disaggregating reaction takes place very quietly and completely if we treat 0·5 grm. of the finely powdered ferro-silicon with 10 grms. of an intimate mixture of carbonate of sodium and potassium (Fresenius's formula, "Qualitative Analysis") with 1 grm. of powdered peroxide of sodium. This is heated slowly before the blowpipe to prevent any projections. The decomposition is soon complete. After cooling on a plate of polished steel, it is treated with boiling water and then with dilute hydrochloric acid in a porcelain crucible. The platinum crucible is well washed and the solution evaporated to dryness on the water-bath, after adding 10 c.c. of nitric acid and 2 grms. of chlorate of potassium. It is then heated in the hot air oven to 110°, and taken up with 20 c.c. of pure hydrochloric acid and 200 c.c. of distilled water. After boiling, it is filtered on the filter-pump, washed with hot water, dried, and calcined in a muffle in a platinum crucible. Perfectly white silica is obtained, $SiO_2 \times 0{\cdot}46667$ = silicon.

This method is most rapid with the very rich ferro-silicons, which are not attacked by acids, and it gives perfectly concordant results.

In the filtrate, the manganese can be estimated by the volumetric method with oxide of zinc and titrated permanganate, and also the sulphur by means of chloride of barium.—*Moniteur Scientifique*, vol. xvi., January, 1902.

CHARACTERISTIC REACTION OF PHENIC ACID.

By M. MANSEAU.

The action of chlorine and bromine on phenols in alkaline solution, especially ammoniacal, is well known.

We further know that the direct oxidation of the phenols gives coloured bodies called quinones, having the property of being generally oxidisable and colourable, and of uniting, by the aid of reducing agents such as sulphurous acid, ferrous chloride, &c., with two atoms of hydrogen, giving *hydroquinones*, $C_6H_4(OH)_2$, and, by incomplete reduction, the *green hydroquinons* or *quinhydrons*,—

$$C_6H_4<\begin{matrix}O-O\\OH-OH\end{matrix}>C_6H_4.$$

It is to an oxidation of this character that we may apparently ascribe the reaction we have obtained while preparing a medicament, well known abroad under the name of "Hagner's olfactive," after a celebrated Polish doctor.

This medicament consists of a mixture of equal parts of carbolic acid, ammonia, tincture of iodine, and camphorated alcohol. It appears to be an excellent remedy for coryza. It suffices to breathe in the volatile principles (ammonia, camphor, and the phenolic ethers) which are given off to obtain rapid relief.

But what is interesting from the chemical point of view is this, that on adding the tincture of iodine to the mixture of carbolic acid and ammonia, a fleeting water-green colouration occurs, quickly disappearing, and leaving an absolutely colourless liquid. We have attempted to fix this reaction and make it persistent. For this purpose we placed, in a test-tube, a few crystals of pure carbolic acid, dissolved in 1 c.c. of alcohol; we then added a few drops of pure ammonia and iodine in alcoholic solution.

The iodine first disappeared very rapidly, then with more difficulty, and finally the solution took a water-green colour, which was persistent in the cold, and even on heating, or on the addition of hydrochloric acid. Nitric and sulphuric acids both destroy or change it. This reaction is absolutely characteristic of phenol, (C_6H_5OH), under the conditions just described.

If we take the phenols most in use, and we consider the diverse reactions produced by the addition of the three substances, alcohol, ammonia, and iodine, in alcoholic solution, we arrive at the following result:—

Different phenols in alcoholic solution:—

Those remaining colourless are—the solutions of phenol (creosote from the beech-tree), thymol, resorcin, naphthol, pyrocatechin, hydroquinone, pyrogallol.

If to these different solutions we add a few drops of pure ammonia, those remaining colourless are—the solutions of phenol (creosote from the beech-tree), thymol, resorcin, and naphthol; those becoming coloured are—the solutions of pyrocatechin, a reddish-brown; hydroquinone, a saffron yellow; pyrogallol, a blackish-brown; orcin, a purple-red, becoming violet.

On the addition of iodine in alcoholic solution to complete absorption, those becoming coloured, though colourless before, are—the solutions of phenol, the characteristic water-green; creosote from beech-trees, a greenish-brown; thymol, flesh coloured, becoming brick-red on the addition of iodine in excess; resorcin, colour of old cognac; naphthol, citron yellow, with the formation of a precipitate of the same colour on the addition of an excess of iodine.

As for the solutions already coloured by the addition of ammonia, on adding iodine that of pyrocatechin becomes cashoo colour without precipitate; that of pyrogallol becomes completely black; that of hydroquinone, saffron-yellow, passing to dark red; that of orcin remains violet.

Gaiacol behaves with this reagent like creosote from the beech, and salicylic acid, which has some analogy with phenic acid with regard to certain reagents, gives a

yellowish-green colour, becoming brown with the formation of a precipitate.

This water-green colour is thus quite characteristic of phenol (C_6H_5OH), and has enabled us to test the purity of white creosotes from coal-tar, sold by druggists and much used by dentists at the present time.

Of four different samples we procured, two consisted of pure phenic acid in solution without traces of *cresylol*; the other two contained this latter product, and, in fact, in this case the water-green colour became yellowish-green, and is easily detected by comparison.

This reaction further enables us to detect phenic acid in phenic water at 1, 2, or 3 per cent. It suffices to put x drops in a tube with x drops of alcohol, add the ammonia, then the iodine until the mixture becomes green.

In creosote from the beech, the greenish-brown colour observed tends to become more green, as the creosote is richer in phenol (C_6H_5OH).

Finally, this reaction is only distinct when we use the reagents in the order we have just described. In the presence of soda or potash we never obtained the water-green colour, but a yellowish tint with the formation of a precipitate, which precipitate is also formed under similar circumstances with the other mono- and di-valent phenols, and thus destroys the sharpness of the reaction to be observed.

It is equally indispensable to operate in alcoholic solution; for in aqueous solution, either with exclusively aqueous solutions of phenic acid or by using iodine water, we obtain precipitates which prevent the reaction, and the decompositions which take place in this case are of quite another nature and so well known that they need not be referred to further.—*Bull. des Trav. Pharm. de Bordeaux*, 1901, p. 117.

THE MECHANISM OF THE ACTION OF PEROXIDE OF HYDROGEN ON PERMANGANIC ACID.

By A. BACH.

To explain the mechanism of the action of peroxide of hydrogen on permanganate of potash in acid solution, two hypotheses have been put forward; that of M. Traube, according to which the reduction of the permanganic acid by the peroxide of hydrogen would be due to the oxidisability of the hydrogen of the latter, and that of M. Berthelot, who endeavours to explain the simultaneous reduction of the peroxide of hydrogen and the permanganic acid by the formation of a trioxide of hydrogen, unstable at the ordinary temperature. While the first hypothesis rests on no experimental basis, the second is founded on the well-known experiment in which M. Berthelot has shown that peroxide of hydrogen decolorises permanganate of potash at a temperature of 12°, without giving rise to any appreciable evolution of oxygen. Up to quite recent times, this experiment was generally considered to be quite conclusive, so that Traube's hypothesis had but few upholders.

Messrs. Baeyer and Villiger (*Berichte*, vol. xxxiii., p. 2488) have recently repeated M. Berthelot's experiment, and found that the decrease in the evolution of oxygen was due, not to the formation of a trioxide of hydrogen stable at a low temperature, but simply to a phenomenon of supersaturation. Consequently, they consider M. Berthelot's hypothesis to be untenable, and revert to that of M. Traube, saying:—"In the present state of our knowledge we regard this manner of viewing the question (that of Traube) as agreeing best with the known facts, and we are of opinion that the authors of elementary text-books would be quite right in adopting it."

In face of this categoric affirmation, I venture to publish a few observations which help to show that, of the two hypotheses in question, it is not that of M. Traube which agrees best with the facts.

I have attempted to approach the question from the experimental side, and that under the following conditions:—

The excellent work recently published by Messrs. Baeyer and Villiger has shown the existence of a series of derivatives of peroxide of hydrogen, of which one—the hydroethylic peroxide, $H.O.O.C_2H_5$—seems to be able to furnish the means of verifying Traube's hypothesis by direct experiment.

We know that peroxide of hydrogen reacts on permanganate of potash in acid solution according to the following equation:—

$$2KMnO_4+3H_2SO_4+5H_2O_2=$$
$$=2MnSO_4+K_2SO_4+8H_2O+5O_2.$$

It is evident that this equation agrees as well with M. Traube's hypothesis as with that of M. Berthelot. But the opposite is the case when we use, instead of peroxide of hydrogen, a mono-substituted peroxide of hydrogen which, like hydroethylic peroxide, $H.O.O.C_2H_5$, contains a radical which is almost if not quite unoxidisable. If Traube's hypothesis is really the correct one, that is to say, if the action of the peroxide of hydrogen on permanganate of potash depends on the oxidation of the hydrogen of the peroxide, then hydroethylic peroxide, which does not contain a single oxidisable atom of hydrogen, should require for titration half as much again permanganate as a solution of peroxide of hydrogen containing the same amount of active oxygen. Consequently, when we determine the active oxygen, in accurately weighed quantities of hydroethylic peroxide, first iodometrically, and then by titration by means of permanganate of potash, this latter operation should show—if Traube's hypothesis is correct—half as much again active oxygen as by the iodometric determination. Naturally it is still necessary to take count of the eventual oxidation of the ethyl group in the hydroethylic peroxide, and to subtract from the amount of permanganate used in the titration the amount corresponding to the oxidation of the ethyl or the products derived from it.

The experiments I undertook with hydroethylic peroxide prepared and purified according to Messrs. Baeyer and Villiger's instructions (*Berichte*, vol. xxxiv., p. 738), and taking every possible precaution, gave almost the same values for the iodometric estimation of the active oxygen and for the titration by means of permanganate of potash.

These experiments thus appear to completely disprove Traube's hypothesis, and afford fresh grounds for upholding that of Berthelot.

But a further study of the reaction has shown that the results obtained cannot be used against the hypothesis of Traube or in favour of that of Berthelot, and for the following reason:—

When we titrate a solution of hydroethylic peroxide by means of a solution of permanganate of potash, we observe that the reaction between these two substances only proceeds, even from the commencement, with extreme slowness. It is only after the lapse of one or two hours that the reduction of the permanganate becomes more active to finally pass through the same stages as in the case of peroxide of hydrogen. It was quite possible to imagine that the sulphate of manganese formed played an important *rôle* in accelerating the reduction of the permanganate by the hydroethylic peroxide. In fact, by adding a certain quantity of sulphate of manganese to the latter, at the commencement of the titration, I found that the progress of the reaction was in every way identical with that which takes place between non-substituted peroxide of hydrogen and permanganate of potash.

It was very probable that, in the presence of sulphate of manganese, the hydroethylic peroxide underwent a hydrolysis, being transformed into peroxide of hydrogen and ethylic alcohol, which would sufficiently explain the fact that the iodometric estimation and the titration of the active oxygen by permanganate of potash gave approximately similar values. The experiments with hydro-

ethylic peroxide did not, therefore, lead to the end I was endeavouring to attain.

I found a better point of comparison in the observation I had occasion to make previously (*Moniteur Scientifique*, 1901, p. 25; *Berichte*, 1900, p. 3111; 1901, p. 1520); that is that the product of the action of concentrated sulphuric acid, or even anhydrous sulphuric acid on dry persulphate of potash, reduced permanganic anhydride, also dissolved in sulphuric acid, concentrated or anhydrous, with great energy, at the same time liberating oxygen.

This product contains a mono-substituted peroxide of hydrogen—mono-persulphuric acid, $HO_3S.O.OH$—which reduces permanganic anhydride under conditions which exclude all possibility of hydrolysis or transformation into peroxide of hydrogen. According to Traube's hypothesis, this product ought to require for titration half as much again permanganic anhydride as a solution of peroxide of hydrogen containing the same quantity of active oxygen.

The experiments previously referred to (*loc. cit.*) were carried out in the following manner:—0·4 grm. of persulphate of potash containing 0·02214 grm. of active oxygen was treated with anhydrous sulphuric acid, the solution was titrated by means of a solution of permanganic acid in anhydrous sulphuric acid, and the oxygen given off was collected over mercury. A solution of peroxide containing the same quantity of active oxygen would have required 8·3 c.c. of permanganic anhydride solution (1 c.c. = 0·002645 grm.), and would set 30·8 c.c. of oxygen at liberty after titration.

Admitting, as is required by Traube's hypothesis, that the atom of hydrogen of the mono-substituted peroxide of hydrogen is oxidised by the permanganic anhydride, we ought to have used 4·15 c.c. of the permanganic anhydride solution for the titration, instead of 8·3 c.c., and equally obtain 30·8 c.c. of oxygen, since, according to Traube, the oxygen set free during the titration comes exclusively from the group —O—O— of the peroxide, of which the hydrogen is oxidised. As a matter of fact, I used 5 c.c. of permanganic anhydride solution for the titration of the product, and collected 24·25 c.c. of oxygen (reduced to 0° and 760 m.m.).

The product, therefore, required *more* permanganic anhydride, and gave *less* oxygen than was expected according to Traube's hypothesis.

This deficit of oxygen (6·55 c.c.) shows plainly that the oxygen liberated by the action of the peroxide of hydrogen by the permanganate of potash does not come exclusively from the group —O—O— of the peroxide, for the quantity of oxygen given off should have been independent of the amount of permanganic anhydride used, and have reached about 30·8 c.c.

As we have seen, the product has required 5 c.c. of permanganic anhydride instead of 8·3 c.c.; that is to say, 3·3 c.c. less than a solution of peroxide of hydrogen containing the same quantity of active oxygen. If, now, we compare the amount of disposable oxygen contained in these unused 3·3 c.c. of permanganic anhydride (0·008728 grm. = 6·05 c.c. O), and the above mentioned deficit of oxygen (6·55 c.c.), we see that the two values agree almost exactly. *The deficit of oxygen is the correlative of the diminution of the quantity of permanganic anhydride used in the titration.**

Contrary to Traube's hypothesis, the oxygen set free by the action of the peroxide of hydrogen on the permanganic acid, there seems to be provided, in equal parts, by the two bodies, each atom of active oxygen of the peroxide uniting with an atom of disposable oxygen of the permanganic acid to form a molecule of oxygen, probably with the intermediate production of trioxide of hydrogen.

If we consider that, in the titration of the product analysed, more permanganic anhydride was used than was necessary for the oxidation of an atom of hydrogen, we are obliged to admit that the only case in which Traube's hypothesis has been put to experimental proof, is decidedly unfavourable to this hypothesis.

On reviewing all the facts relative to the action of peroxide of hydrogen on permanganic acid, we are bound to arrive at the conclusion that Berthelot's hypothesis is much more plausible and rational than that put forward by Traube. Even admitting that the existence of trioxide of hydrogen is not proved by M. Berthelot's experiment, it nevertheless does not follow that his hypothesis is a false one. We know of a number of reactions in which there is a formation of intermediary products, which up to the present have not been isolated. As in every other respect M. Berthelot's hypothesis seems to be free from objection—this certainly cannot be said of Traube's*—it is not easy to see why the latter should be preferred to the former. In any case, neither of these two hypotheses is yet fit to be included in elementary text-books.—*Moniteur Scientifique*, Series 4, vol. xvi., January, 1902.

* The cause of this diminution is probably due to the formation of a superoxygenated peracid—a derivative of tetroxide of hydrogen—the consequence of which is to render inactive a portion of the active oxygen, since the atoms of this latter unite in groups of three to form compounds containing, like ozone, a chain of three atoms of oxygen, of which only one is active.

* To admit, as is done by Traube, that peroxide of hydrogen contains easily oxidisable hydrogen, *united directly* with active oxygen, seems contrary to reason.

CHEMICAL SOCIETIES OF THE NINETEENTH CENTURY.†

By HENRY CARRINGTON BOLTON, Ph.D.

The beginning of a new century affords an opportune period for chronicling the progress of chemistry as shown by the organisations formed to foster its study and to stimulate its adherents. In the following pages an attempt has been made to place on record the statistics of the Chemical Societies of the World for the year 1900, and to indicate those that ended their careers within the nineteenth century. The data have been obtained chiefly by correspondence, and thanks are due to the officers of societies who have responded to inquiries. I am also under special obligations to Dr. Paul Dorveaux, Librarian of the Ecole Supérieure de Pharmacie, Paris; to Professor Bohuslav Brauner, of the Bohemian University, Prague; to Professor George W. A. Kahlbaum, of the University of Basel; and particularly to the Smithsonian Institution, for aid in securing the information sought.

The fact that chemical societies were organised and in operation in the United States of America long before they existed in Europe has been shown in my paper, "Early American Chemical Societies," read to the Chemical Society of Washington, April 8, 1897. The two pioneers in this field were the Chemical Society of Philadelphia, founded in 1792, and the Columbian Chemical Society of Philadelphia, founded in 1811. Of these, some particulars will be found in their proper order.

In the following list the societies are placed in chronological order under each country, and the countries are arranged alphabetically. Of each society the following data are given so far as attainable:—

Seat, and date of founding.
Name of President, and membership in 1900. (No deductions have been made for duplication).
Serial publications. (For full details consult "A Select Bibliography of Chemistry," by Henry Carrington Bolton, Washington, 1893-1899, 3 vols., 8vo).
Remarks.

The results of this census are given in the following table:—

† Read at the Twenty-fifth Anniversary of the American Chemical Society, held in New York City, April 12—13, 1901. From advance proofs of the *Smithsonian Miscellaneous Collections*, 1901.

CHEMICAL SOCIETIES OF THE WORLD.

Membership in 1900.

Country.	No. of societies.	No. of members.
Austria	7	3072
Belgium	3	740
France	10	4065
Germany	10	7559
Great Britain	9	7550
Italy	5	479
Japan	2	1012
Russia	1	327
South Africa	1	(?)
Switzerland	2	94
United States America	5	2379
Victoria	1	100
Totals	56	27,377

AUSTRIA.

CENTRALVEREIN FÜR RÜBENZUCKER INDUSTRIE IN DER OESTERREICHISCH-UNGARISCHEN MONARCHIE.

Founded at Vienna in 1854. In 1900: President, August Freiherr von Stummer; members, 213; associates, 49.

Publications.—Organ des Vereins für R.-I., 1863-1874; Organ des Centralvereins für R.-I., 1875-1887; Oesterreichisch-ungarische Zeitschrift für Zucker-Industrie und Landwirthschaft, 1888-1900. (Beilagen). — Der Marktbericht, 1874-1885; Wochenschrift des Centralvereins für Rübenzucker in der oesterreichisch-ungarischen Monarchie, 1886-1900.

NOTE.—The Society maintains a Chemical Experiment Station under the direction of F. Stohmer.

VEREIN ZUR HEBUNG DER ZUCKERFABRIKATION IM KÖNIGREICH BÖHMEN. (Also with a Bohemian name).

Founded at Prag in 1868 under the presidency of Ferdinand Urbánek; the languages used were Bohemian and German. It was disbanded in 1874.

Publication.—Zeitschrift für Zucker-Industrie. Organ des Vereins. Prag. 3 vols., 1872-74.

NOTE.—This journal is not to be confounded with *Zeitschrift für Zucker-Industrie in Böhmen*, established at Prag in 1877 and current.

CHEMICKA SPOLEČNOST: SPOLEK ČESKÝCH CHEMIKU. (Chemical Society: Union of Bohemian Chemists).

Founded at Prag in 1872. In 1900: President, K. Preis; honorary members, 11; active members, 318; correspondents, 77.

Publications.—Zprávy spolku českých chemiku. 2 vols., 1872-76. (Reports). Listy Chemické, 1877-1900. (Letters).

NOTE.—The Society has also published a Chemická Knihovna (Chemical Library) in 8 vols.

ZEMSKY SPOLEK PRO PRUMYSL CUKROVARNICKY V ČECHÁCH; VEREIN DER ZUCKERINDUSTRIE IM KÖNIGREICH BÖHMEN.

Founded at Prag in 1876. In 1900: President, Gustav Hodek; members, 325.

Publications.—Zeitschrift für Zucker-Industrie in Böhmen, 1877-1900. (Beilage).—Prager Zuckermarkt, 1881-1900.

OESTERREICHISCHE GESELLSCHAFT ZUR FÖRDERUNG DER CHEMISCHEN INDUSTRIE.

Founded in Prag in 1878. In 1900: President, Georg Zetter; honorary members, 3; members, 196.

Publications.—Bericht der oesterreichischen Gesellschaft zur Förderung der chemischen Industrie, 1879-98. Since 1899 the organ of the Society is Oesterreichische Chemiker-Zeitung.

SPOLECNOST PRO PRUMYSL CHEMICKÝ. (Society of Chemical Industry).

Founded in 1892 at Prag. In 1900: President, J. B. Lambl; honorary members, 20; active members, 440; correspondents, 54; founders, 57.

Publications.—The organ of the Society since 1892 is Casopis pro prumysl chemicky, of Prag, which had been established in 1891. The Society has also published three volumes of a technological library, Knihovna technologicko chemická.

WIENER VEREIN ZUR FÖRDERUNG DES PHYSIKALISCHEN UND CHEMISCHEN UNTERRICHTS.

Founded in 1895 at Vienna. In 1900: President, Victor von Lang; members, 317.

Publication.—Vierteljahrsberichte der Wiener Verein zur Förderung des physikalischen und chemischen Unterrichts, 1895-1900.

VEREIN OESTERREICHISCHER CHEMIKER IN WIEN.

Founded in 1897 at Vienna. In 1900: President, J. Klaudy; members, 878; founders, 14.

Publication.—Oesterreichische Chemiker Zeitung, 1898-1900.

BELGIUM.

ASSOCIATION BELGE DES CHIMISTES.

Founded August 4, 1887, at Brussels. In 1900: President, L. L. de Koninck; honorary members, 4; active members, 482; associates, 21; correspondents, 8.

Publication.—Bulletin de l'Association Belge des chimistes, 1887-1900.

NOTE.—The Association has eight sections, viz., Liége, Louvain, Gembloux, Charleroi, Mons, Gans, Antwerp, Brussels.

SOCIÉTÉ TECHNIQUE ET CHIMIQUE DE SUCRERIE DE BELGIQUE.

Founded February 26, 1896, at Brussels. In 1900: President, Eugène Meeus; members, 173; patron, 1.

Publications.—La sucrerie Belge, which was established August 31, 1872, has been the organ of the Society since its foundation. The Society has also published several pamphlets on technical topics.

NOTE.—The formation of Sections was under discussion in 1900.

SYNDICAT DES CHIMISTES PUBLICS DE BELGIQUE.

Founded in 1897 at Brussels. In 1900: President, François Sachs; members, 51.

Publication.—Bulletin du Syndicat des Chimistes publics de Belgique, 1897-1900.

NOTE.—The Society has in preparation "Recueil générale des méthodes d'analyse usitée dans les laboratoires publics."

SOCIÉTÉ GÉNÉRALE DES FABRICANTS DE SUCRE DE BELGIQUE is not a chemical society; its organ is *La sucrerie Belge*, 1872-1900.

(To be continued).

Action of Organo-magnesium Compounds on β-Ketonic Ethers.—V. Grignard.—The author's researches on this subject show that, in presence of organo-magnesium compounds, (1) acetylacetic ether reacts only in its enolic form; (2) its monoalcoyl derivatives are really mixtures of the ketonic and enolic forms; (3) the products of condensation of the aldehydes with acetoacetic ether possess Claisen's formula.—*Comptes Rendus*, CXXXIV., No. 15.

A NEW INVESTIGATION CONCERNING THE ATOMIC WEIGHT OF URANIUM.*

By THEODORE WILLIAM RICHARDS
and
BENJAMIN SHORES MERIGOLD.

(Continued from p. 209).

Methods of Analysis.

By the use of these devices we were able to prepare and weigh pure uranous bromide in a definite state. There still remained, however, the problem of devising a suitable method of analysis. As previously mentioned, all uranous compounds reduce silver nitrate, making impossible the usual method of procedure in halogen determinations.

The method of precipitating the uranium and determining bromine in the filtrate involves too much danger of loss of material in the multiplicity of operations. The most satisfactory solution of the problem seemed to be to oxidise the compound to the uranyl salt, provided this could be done without loss of bromine. Nitric acid is of course effective as an oxidising agent, but the oxidation is accompanied by loss of bromine. After much experimenting, hydrogen dioxide was found to be the most suitable oxidiser. From neutral solutions of uranium compounds, hydrogen dioxide precipitates a hydrated peroxide of uranium. If the solution is slightly acid, this precipitation is prevented, and the uranous compound completely oxidised to the uranyl state. The weighed sample of uranous bromide was dissolved in considerable water—at least 400 c.c. of water to each grm. of bromide. The bottle containing the bromide was opened by means of a suitable glass fork, either below the water or just above the surface, so that it could be instantly submerged, and thus avoid loss of hydrobromic acid by the action of moist air. The calculated volume of a standard solution of pure hydrogen dioxide was then diluted to about 100 c.c., 1 c.c. of pure dilute sulphuric acid was added, and the mixture was slowly run into the solution of uranous bromide. The green colour of the uranous salt soon changes to the yellow colour characteristic of uranyl compounds. On adding the first few c.c. of the dilute hydrogen dioxide solution, a greenish-white precipitate came down. Addition of more of the acid dioxide solution re-dissolved it, and the resulting solution was perfectly clear. This peculiar hydrolytic action is due to the acid, and not to the hydric dioxide, for the same reaction occurs if dilute sulphuric acid alone is added to the solution.

The explanation of this interesting phenomenon, which is just the opposite of what might have been expected, is, undoubtedly, that the bromide is already hydrolysed to a great extent by merely dissolving in water. The hydrate is probably in solution in the colloidal state. Evidence of this is found in the fact that if the clear aqueous solution of uranous bromide is allowed to stand exposed to the air, a hydrate gradually separates, giving to the solution a cloudy, murky appearance. After two or three days this precipitate disappears, giving place to a clear yellow solution of oxybromide and hydrobromic acid. The addition of sulphuric acid coagulates the colloid before it can all be converted into uranyl salt.

In order to be sure that no bromine or hydrobromic acid is lost by this method of oxidation, the following experiment was made:—0·5 grm. of bromide was dissolved in 250 c.c. of water, 50 c.c. of dilute sulphuric acid (1 : 10) was added, and the hydrogen dioxide solution was run in. This was done in a closed flask, similar in construction to a gas washing bottle. A current of air was drawn through the bottle and then through starch solution containing potassium iodide to see if bromine is liberated. Not the slightest trace of blue colour appeared in the starch solution, even after adding a large excess of hydrogen peroxide, and allowing it to stand over night. A test for hybrobromic acid was sought in a similar way, by drawing the air through a solution of silver nitrate, again with negative results, as was to have been expected. These experiments show conclusively that uranous bromide can be oxidised completely by hydrogen dioxide, without loss of bromine.

Silver nitrate, in moderately concentrated solutions is not acted upon by a 3 per cent solution of hydrogen peroxide. Consequently, a considerable excess of the latter reagent could do no harm. Nevertheless, care was taken never to add more than the calculated amount of hydrogen dioxide. Moreover, the solution of hydrogen dioxide used contained only 1 per cent of this reagent, and this was diluted ten times before adding to the bromide solution, thus reducing to a minimum the possibility of too vigorous oxidation, with consequent liberation of bromine.

After the oxidation, bromine was precipitated by pure silver nitrate in the usual manner. This precipitation was conducted in an Erlenmeyer flask fitted with a glass ground stopper. The silver bromide was collected on a Gooch crucible, and dried in an electrically heated drying oven. Of course the asbestos shreds carried away in washing the silver bromide were collected by passing the filtrate and wash water through a fine filter, and their weight was added to that of the silver bromide. The bromine determination was carried on in orange-coloured light.

It was found in the work upon cobalt and nickel that the porcelain tube is attacked by bromine vapour at the high temperature employed during the sublimation, with the result that sodium bromide was always present in the sublimate. In these investigations this impurity was determined by the reduction of the bromide to the spongy metallic state by means of hydrogen and extraction by water (*Proc. Amer. Acad. Arts Sci.*, 1899, xxxiv., 329, 359). A somewhat similar method was tried with uranium. Since hydrogen reduces uranous bromide only to the tribromide, the bromide was ignited in a current of air, and the resulting oxide leached with water. It was found to be impossible to oxidise the bromide completely. A little uranous bromide invariably remained, and was washed out with the alkali. Both dry and moist air was tried, also ignition in steam, but in every case uranium was washed out in considerable quantity.

Precipitation of the uranium by hydrogen dioxide was next tried, but it was found impossible to precipitate the uranium completely. The rather unsatisfactory method of determining the sodium in the filtrate from the bromine precipitation, or in a new sample of uranous bromide as nearly as possible, after removing the uranium with ammonium sulphide, appeared to be the only available method. The filtrate and wash waters from the bromine precipitation were evaporated in platinum to small bulk, and the uranium and excess of silver precipitated by pure colourless ammonium sulphide. This reagent precipitates uranium completely. The filtrate was then evaporated to dryness, the ammonium salts expelled by ignition, and the residual sodic nitrate converted to the sulphate, and weighed as such. Of course these operations were all conducted in platinum vessels. This method of work is not wholly satisfactory, on account of the complexity of operations involved, but it seems to be the only practical method.

Purification of Materials.

As the source of uranium, commercial "chemically pure" uranium acetate was used.* This was first converted to the chloride on account of the greater solubility of this compound—by precipitation as ammonium uranate, and re-dissolving in dilute hydrochloric acid. To the hot and slightly acid solution, pure sulphuretted hydrogen was added to saturation. The free acid was then neutralised

* Contributions from the Chemical Laboratory of Harvard College. From the *Proceedings of the American Academy of Arts and Sciences*, xxxvii., No. 14.

* This method of uranium purification, with some modifications and additions, is similar to that employed by Zimmermann (*Ann. der Chem. u. Pharm.*, ccxxxii., 299).

with ammonic hydroxide, a slight excess of the alkali was added, and more sulphuretted hydrogen was run in. In this way some uranyl sulphide was precipitated, in order to sweep down with it any colloidal sulphides of the higher groups which might otherwise escape removal. The excess of sulphuretted hydrogen was boiled off, and after standing over night the supernatant liquid was decanted through a washed filter.

The next step depended upon the fact that uranium remains in a solution of the double carbonate of ammonium and uranium, in the presence of an excess of ammonium sulphide, while all the other members of the aluminum and iron groups are thrown down by this reagent. Consequently, ammonic hydrate and ammonium carbonate in slight excess were added to the filtrate, forming the double carbonate. If the solutions are concentrated, the double carbonate is precipitated when more than a slight excess of ammonic carbonate is used. This happened in some cases, when it was necessary to re-dissolve the precipitate in dilute hydrochloric acid, and again add ammonic carbonate in more dilute solution. About 50 grms. of carbonate per litre was found to give the best results. Ammonic hydroxide was then added to the hot solution, and sulphuretted hydrogen in excess. After standing over night the solution was filtered. In several of the more concentrated solutions, a considerable quantity of the salt crystallised out. These crystals were worked up separately, as they were probably purer than the solution. On boiling the solution to decompose the excess of ammonium sulphide, some of the ammonic carbonate was decomposed, causing the precipitation of some uranium sulphide. This precipitate was discarded, as it might have contained iron, or other analogous metals which had previously escaped precipitation. Dilute hydrochloric acid in slight excess was added, and the carbon dioxide was expelled by boiling. The free acid was then almost neutralised with pure ammonic hydroxide, and pure ammonic sulphydrate added in excess. The colour of the resulting precipitate of uranium sulphide varies greatly with the temperature. In warm solution it was at first reddish-brown, while that precipitated in the cold varied from bright red to brownish-yellow. On washing, all turn black, the sulphide being decomposed into uranous oxide and sulphur. After thorough washing the resulting mixture of oxide and sulphur was ignited in a porcelain dish, the green urano-uranic oxide being the product.

The oxide was then dissolved in a platinum dish in redistilled nitric acid, evaporated and re-crystallised from nitric acid solution. Uranyl nitrate does not crystallise well from aqueous solution, but it was found that if a little nitric acid is added, it crystallises readily in fairly large monoclinic prisms. This re-crystallisation was repeated ten times from acid solution, and, finally, twice from aqueous solution. Finally, the pure nitrate was converted to the oxide by ignition in platinum. A second sample, used in the preliminary series, was prepared by repeated fractionation of the mother-liquors of the first sample.

Since this work was carried out, Sir William Crookes (*Proc. Roy. Soc*, 1900, lxvi., 409) has published the account of several methods by which he was able to prepare specimens of uranyl nitrate which were not radio-active. The radio-activity of uranium has hitherto been supposed to be characteristic of this element. Crookes has shown, however, that this is not the case, but that the active element can be separated by treatment with ether, by fractional crystallisation, or by treatment with excess of ammonium carbonate. Unfortunately, none of the pure oxide prepared for this investigation remained, hence it is impossible to test directly its radio-activity. Since two of Crookes's method were used in purifying our material, viz., the ammonium carbonate treatment and fractional crystallisation, it is highly improbable that our oxide was radio-active. In repeating Crookes's work with nitrate made from some of the same material used in preparing our best nitrate, it was found that a sample of the *fifth* crystallisation gave no trace of action on twenty-four hours' exposure to a quick photographic plate. The material used in this experiment had not been submitted to the ammonium carbonate treatment. When it is considered that the material used for our atomic weight determinations was first put through the carbonate process—in itself sufficient to remove the radio-active element—and then was re-crystallised *twelve times* as nitrate, it would seem that our pure oxide must have been free from all radio-active material.

There is another phase of this subject that deserves to be considered, namely, the possible effect of radio-active matter, even if present, upon the atomic weight value. The purest specimen of radium or "polonium" yet obtained has consisted of a mixture containing probably little more than 50 per cent of the active element, as nearly as could be estimated. This highly impure material, however, possesses 8000 times the radio-activity of uranium. The radio-active power of the pure material is undoubtedly very much greater than that of the impure mixture. Consequently, the quantity of radio-active substance necessary to give to uranium the comparatively slight degree of activity that it possesses must be exceedingly minute. Giesel has recently shown (*Berichte*, 1900, xxxiii., 3569) that a quantity of radium so small that it cannot be detected by sulphuric acid is sufficient to affect a photographic plate. Crookes also says on this point:—"Considering my most active UrX does not contain sufficient of the real material to show in the spectrograph, yet is powerful enough to give a good impression on a photographic plate in five minutes, what must be its dilution in compounds which require an hour, a day, or a week to give an action?" (*Proc. Roy. Soc.*, 1900, lxvi., 422). Even in the ordinary active uranium compounds it is most unlikely that the active element—if indeed it is an element—could possibly be present in quantity sufficient to exert any influence whatever upon the atomic weight of uranium.

Pure carbon was obtained by ignition of sugar. Large, clear crystals of the best "rock candy" of commerce were were ground up in a porcelain mortar, and ignited at low heat in a platinum dish as long as organic gases were given off. The resulting charcoal was then powdered in an agate mortar, and ignited in a hard glass combustion tube; first in a stream of pure, dry nitrogen, and finally in a stream of bromine vapour. In this way the carbon was freed from any impurities which might, if present, be acted upon during the sublimation, and contaminate the sublimate. Owing to the presence of undecomposed carbohydrates, or possibly of water, most of the bromine was converted into hydrobromic acid. Heating in bromine was continued until acid fumes ceased to be given off. Finally, the carbon was again heated in a current of dry nitrogen. Five grms. of carbon, thus prepared, left no visible or weighable residue after combustion in oxygen.

The method of bromine purification was essentially identical with that used in many other atomic weight investigations in this laboratory, and has been proved by long experience to be the most efficient and satisfactory. Commercial, "pure" bromine was partially freed from chlorine by shaking with a 15 per cent solution of potassic bromide. One-fourth of the bromine was then converted to calcic bromide by running it slowly into milk of lime in the presence of a large excess of ammonia. The calcic bromide solution was filtered and concentrated by evaporation, and the rest of the bromine was added to it. A little zinc oxide was then added, and after standing over night the bromine was distilled, nearly free from chlorine. Most of the iodine is removed as zinc iodate. After redistilling the bromine, in order to remove any calcium bromide that may have spattered over in the first distillation, it was converted into hydrobromic acid by slowly dropping it into a mixture of red phosphorus and hydrobromic acid. The red phosphorus was at first washed free from chlorides. The hydrobromic acid, containing some free bromine, was distilled. The free bromine liberates any iodine which may have escaped the zinc oxide. The first portion of

the distillate, containing free bromine and iodine, and organic matter, was rejected, and so was the last portion, which may have contained traces of arsenic. The hydrobromic acid was then converted into bromine by distilling over pure manganese dioxide previously treated with sulphuric acid and washed. One-half the bromine is obtained by the manganese dioxide alone. As soon as no more bromine comes off, a little re-distilled sulphuric acid is added, and the rest of the bromine was obtained. It was then re-distilled several times, rejecting the first and last portions, and finally dried over pure phosphorous pentoxide.

The silver precipitation also presents no new features, except, perhaps, its somewhat unusual thoroughness. Partially purified silver was dissolved in nitric acid, diluted, and precipitated with pure hydrochloric acid. After thorough washing the chloride was reduced by invert sugar and sodic hydrate which had been purified by electrolysis. The metallic silver was thoroughly washed, dissolved in nitric acid, and again precipitated as chloride, and reduced. It was then dried and fused on charcoal; the lumps of silver were cleaned with sand, dissolved in pure nitric acid, diluted to a volume of two litres, and again precipitated with pure hydrochloric acid. The resulting chloride was then digested on the steam bath with aqua regia, washed, and once more reduced by invert sugar and sodic hydrate. After drying, it was fused on pure sugar charcoal. The buttons of silver were cleaned with sand, and then purified electrolytically, a small portion being dissolved in nitric acid to serve as the electrolyte, and the rest serving as anode material. The crystals of electrolytic silver were then dried over potash, and fused *in vacuo* on a boat of pure lime. The buttons of silver thus obtained were treated with nitric acid to remove the surface, dried, and kept over potash. A second sample was obtained by fusing *in vacuo* electrolytic silver which had been prepared from the silver bromide obtained in Dr. Baxter's work upon cobalt, which was known to be very pure.

Hydric dioxide was purified as follows:—To a solution of the ordinary commercial peroxide prepared for medicinal use, was added a solution of baric hydroxide, which had been purified by re-crystallisation. The precipitated baric dioxide was washed until a nitric acid solution of the same showed no trace of halogen. It was then added to pure dilute sulphuric acid, and the resulting solution of hydric dioxide was filtered and distilled in a partial vacuum. The solution thus obtained showed no trace of halogen, and left no visible residue on evaporation in platinum.

Ammonium sulphide was made from pure ammonia, which had been re-distilled in platinum, and pure sulphuretted hydrogen. It left no visible residue on evaporation in platinum.

Hydrochloric and nitric acids were re-distilled in a platinum still, and throughout the work platinum vessels were used wherever possible.

Water was twice re-distilled, once over alkaline potassic permanganate, and again over acid potassic sulphate from a Jena glass flask, a block-tin condenser and Jena glass receiver being used.

(To be continued).

The Mutual Reaction of Arsenious Acid and Permanganate.—O. Kühling.—The estimation of arsenious acid can be effected in a very satisfactory manner by means of a titrated solution of permanganate, proceeding in the same way as for phosphorous acid (*Berichte*, vol. xxxiii., p. 2914). As in this latter case, we can use sulphate of zinc to facilitate the oxidising action of the permanganate, but it is still more simple to use a 10 to 20 per cent solution of sulphuric acid. The operation should be carried out in a nearly boiling solution, and it is best to add almost the whole of the permanganate necessary for the oxidation at one time.—*Berichte*, vol. xxxiv., p. 404.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, Thursday, April 17th, 1902.

Prof. TILDEN, D.Sc., F.R.S., Treasurer, in the Chair.

MESSRS. Davis, Dorée, Aders Plimmer, Allworthy, and Bedwell were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. William Thomas Clough, 3, Watford Villas, Battersea Park, S.W.; Henry Wilson Davis, 18, Crescent Road, Kingston Hill, Surrey; Thomas Butterworth Hallowell, New Mills, Derbyshire; Charles B. Lessner, 46, Broadfield Road, Hither Green, S.E.; Thomas Henry Moore, 19, Sandmere Road, Clapham, S.W.; Prafulla Chandra Rây, Calcutta, India; William Pearson Skertchly, 11, Billiter Square, E.C.; Hector Stewart, 479, Collins Street, Melbourne; William John Wells, Springburn, Blackburn; Joseph West, 50, Poplar Grove, Fenton, Stoke-on-Trent.

Of the following papers, those marked * were read:—

*56. "*Dimercurammonium Nitrite and its Haloid Derivatives.*" By P. C. RAY.

Some account has already been given (*Proc.*, 1901, xvii., 96) of the investigation which is here more fully and accurately described. Mercurous nitrite partly decomposes in dissolving in water, giving metallic mercury and a solution of both mercuric and mercurous nitrites (*Trans.*, 1897, lxxi., 337). Adding to this solution just enough sodium chloride and filtering, there is left a solution of mercuric sodium nitrite, which gives, with excess of ammonia, a white precipitate of a new salt, $2NHg_2NO_3,H_2O$, dimercurammonium nitrite. On dissolving it in hydrochloric acid and evaporating, there is left a new mercuric ammonium chloride, $2HgCl_2,NH_4Cl$, a white, crystalline, volatile, and fusible salt. The corresponding bromide can be obtained in a similar way, as well as by evaporating a hydrobromic acid solution of the single bromides in the right proportions; it is largely decomposed by water into its component salts. With potassium hydroxide, not in excess, these double salts give dimercurammonium chloride and bromide, $2NHg_2Cl,HgCl_2$ and $2NHg_2Br,HgBr_2$, the former not quite pure. With excess of potassium hydroxide, they yield the half-hydrated dimercurammonium chloride and bromide, $2NHg_2Cl,H_2O$ and $2NHg_2Br,H_2O$, the former already described by André.

When either dimercurammonium chloride or bromide is dissolved in hydrobromic acid, the above formulated mercuric ammonium bromide is obtained on evaporation; when dimercurammonium bromide is dissolved in hydrochloric acid, a mercuric ammonium chlorobromide, $2HgCl_2,NH_4Br$, is left on evaporation. From this chlorobromide, potassium hydroxide reproduces quantitatively dimercurammonium bromide, a fact which led the author at first to regard the chlorobromide as dimercurammonium bromide in union with 4 molecules of hydrochloric acid, and the corresponding double chloride and double bromide as $NHg_2Cl,4HCl$, and $NHg_2Br,4HBr$, respectively.

The author considers that the chemical composition of dimercurammonium nitrite and its derivatives gives much support to the Rammelsberg-Pesci theory of the dimercurammonium constitution of all ammoniated mercury salts.

*57 "*Preparation and Properties of 4-iso*Propyldihydroresorcin.*" A Correction. By A. W. CROSSLEY.

The nomenclature adopted in the preliminary notice of this work (*Proc.*, 1901, xvii., 172) has been changed. The substance there spoken of as 2 : 6-diketo-4-*iso*propylhexamethylene is now called 4-iso*propyldihydroresorcin*, for though this and other similar substituted dihydroresorcins behave as diketones towards hydroxylamine, there can be no doubt that their most usual form is the keto-enol modification represented by the following formula:—

$$\begin{matrix} CH_2{\cdot}CO \\ (CH_3)_2CH{\cdot}\dot{C}H\ \dot{C}H \\ \dot{C}H_2{\cdot}\ddot{C}OH \end{matrix}$$

*58. "*Oxonium Salts of Fluoran and its Derivatives.*" By J. T. HEWITT and J. N. TERVET.

Fluoran (the phenolphthaleinanhydride of Baeyer) and some of its derivatives have been examined as to their power of forming salts with strong mineral acids. The following salts have been prepared and examined:—*Fluoran nitrate*, $C_{20}H_{12}O_3,HNO_3$; *monosulphate*, $C_{20}H_{12}O_3,H_2SO_4$; *dimethylfluoran nitrate*, $C_{22}H_{16}O_3,HNO_3$; *dimethylfluoran monosulphate*, $C_{22}H_{16}O_3,H_2SO_4$; *dimethylfluoran disulphate*, $C_{22}H_{16}O_3,2H_2SO_4$; *fluorescein hydrochloride*, $C_{20}H_{12}O_5,HCl$; *fluorescein monosulphate*, $C_{20}H_{12}O_5,H_2SO_4$; *fluorescein disulphate*, $C_{20}H_{12}O_5,2H_2SO_4$.

*59. "*Influence of Substitution on the Reactivity of the Aromatic Metadiamines.*" By G. T. MORGAN, D.Sc.

The successive introduction of methyl groups into the three positions adjacent to the nitrogen atoms of *m*-phenylenediamine is attended by a well-marked diminution in the reactivity of the diamine towards methylating agents, the action of methyl chloride or bromide being prevented when the substitution of the three hydrogen atoms is complete.

m-Tolylenediamine (2 : 4-diaminotoluene), like *m*-phenylenediamine, gives rise to a mixture of tertiary base and quaternary salt; 4 : 6-diamino-*m*-xylene yields the tertiary base as the sole product, whilst diaminomesitylene is not affected.

These results agree with those obtained by Pinnow (*Ber.*, 1897, xxx., 3110; 1899, xxxii., 1401; 1901, xxxiv., 1129), and also by E. Fischer and Windaus (*Ber.*, 1900, xxxiii., 345 and 1967).

The tertiary diamines containing one free para-ortho-position with respect to nitrogen are very reactive substances, darkening on exposure, yielding unstable platinichlorides, and interacting readily with formaldehyde and the diazonium salts.

4 : 6-*Tetramethyldiamino-m-xylene*, which contains methyl groups in each of the para-ortho-positions, is a comparatively inert substance which is not darkened by exposure to light or air; its *platinichloride* is stable, and the base itself does not condense either with formaldehyde or diazonium salts. This diamine boils at 243—245° under 757 m.m., and at 124—125° under 12 m.m. pressure; its *picrate* crystallises in yellow prisms melting when suddenly heated at 202—203°, but decomposing at 193—195° when maintained at this temperature for some time.

2 : 4-*Tetramethyldiaminotoluene* is a pale brownish-yellow oil boiling at 254—256° under 757 m.m., and at 148—150° under 24—26 m.m. pressure; its sp. gr. is 0·9661 at 24°; its *platinichloride* is decomposed by hot water; the *picrate* crystallises from alcohol in yellow prisms melting at 162—163°. The *methobromide*, $NMe_2{\cdot}C_6H_3(CH_3)NMe_3{\cdot}Br$, produced together with the tertiary diamine in the methylation of *m*-tolylenediamine, yields a *platinichloride*, $NMe_2{\cdot}C_6H_3(CH_3)NMe_3,HPtCl_6$, crystallising in brownish-yellow scales.

p-Nitrobenzene-5-azo-2 : 4-tetramethyldiaminotoluene, resulting from the action of *p*-nitrobenzenediazonium chloride on 2 : 4-tetramethyldiaminotoluene, crystallises from alcohol or ethyl acetate in dark green leaflets with a bronze reflex, and melts at 126—127°.

2 : 4 : 2' : 4'-*Octamethyltetraminoditolyl-5 : 5'-methane*, prepared by condensing 2 : 4-tetramethyldiaminotoluene with formaldehyde, crystallises from alcohol in colourless prisms melting at 86°; its *picrate* separates in spherical aggregates of yellow crystals, and melts at 147—148°.

60. "*The Influence of certain Acidic Oxides on the Specific Rotations of Lactic Acid and Potassium Lactate.*" By G. G. HENDERSON and D. PRENTICE, Ph.D.

The authors have examined the influence of antimonious, arsenious, and boric oxides on the rotations of aqueous solutions of potassium lactate and lactic acid, with the object of ascertaining whether any compounds of the tartar emetic type are formed in solution by interaction of these substances.

It was found that antimonious oxide is almost insoluble in solutions of potassium lactate, and that the rotations of the solutions are not appreciably affected by the presence of traces of antimonious oxide. Arsenious oxide and boric acid, on the other hand, were found to dissolve readily in aqueous solutions of potassium lactate and lactic acid. The rotations of the solutions are altered by the presence of the dissolved oxide, and in each case the maximum change occurs when the substances are present in the proportions requisite for the formation of an arseniolactate, $(AsO)C_3H_4O_3K$, and a borolactate, $(BO)C_3H_4O_3K$, respectively. The rotation of a solution of lactic acid is slightly diminished when arsenious oxide is dissolved in it, but considerably increased by the presence of boric acid.

Experiments with molybdic and other oxides are in progress.

61. "*The Amounts of Nitrogen as Ammonia and as Nitric Acid, and of Chlorine in the Rain-water Collected at Rothamsted. A Report to the Lawes Trust Committee.*" By N. H. J. MILLER.

Results of monthly determinations of nitrogen as ammonia and as nitrates in the rain-water collected during thirteen harvest years (September, 1888, to August, 1901) at Rothamsted showed that the total nitrogen in these forms varied from 3·31 lbs. to 4·43 lbs. per acre per annum. The average amounts for the winter and summer months, and for the whole year are as follows:—

	Rainfall (inches).	Nitrogen per million as ammonia.	Nitrogen per million as nitrates.	Nitrogen per acre (lbs.) as ammonia.	Nitrogen per acre (lbs.) as nitrates.	Total.
Winter.	14·32	0·381	0·175	1·233	0·568	1·801
Summer	12·93	0·506	0·191	1·479	0·560	2·039
Year ..	27·25	0·440	0·183	2·712	1·128	3·840

Of the total nitrogen 70 per cent is present as ammonia and 30 per cent as nitrates. During the summer months there is an increased production of ammonia, whilst the amount of nitric nitrogen (per acre) is nearly the same in summer as in winter.

Chlorine has been determined monthly for twenty-four years. The average amounts are as follows:—

Sept. 1877 to Aug. 1901.	Rainfall (inches).	Chlorine per million.	Chlorine per acre (lbs.).
Winter months . ..	14·65	3·05	10·12
Summer months ..	14·13	1·49	4·75
Year	28·78	2·28	14·87

The yearly amounts of chlorine in the rain vary considerably (maximum 21·19, minimum 10·32 lbs. per acre). The variations depend less on the total rainfall for the year than on the amount of rain during the winter months.

The rain falling at Rothamsted supplies, not only sufficient chlorine, but also enough sulphuric acid for the requirements of most crops.

62. "*The Amounts of Nitrogen, as Nitrates, and Chlorine in the Drainage through Uncropped and Unmanured Land. A Report to the Lawes Trust Committee.*" By N. H. J. MILLER.

The percolation through the 20, 40, and 60 inches of soil in the three Barnfield drain gauges amounted during 24 harvest years, Sept. 1877 to Aug. 1901, to 14·39, 15·30, and 14·41 inches per annum, being 50·0, 53·2, and 50·1 per cent respectively of the rainfall for the same period. The maximum and minimum drainage occur in November and June.

The average loss of nitrogen, as nitrates, in the drainage is more than 30 lbs. per acre per annum, and about half of this loss takes place during October, November, and December. The yearly losses of nitrogen differ widely, according to the amount and distribution of the rain and drainage. This makes it difficult to form a decided opinion as to whether nitrification is less active at

the present time than in earlier years. A very decided falling off is perhaps not to be expected in the near future. The soil of the 20-inch gauge is estimated to have contained, in 1870, as much as 6000 lbs. of nitrogen per acre, and of this amount only about 15 per cent has been found in the drainage; in the case of the 60-inch gauge, the estimated loss is only 6·5 per cent of the total initial nitrogen of the soil. In addition to loss of nitrogen, there is, however, a considerable loss of lime, amounting to 11 lbs. per acre (or about 20 lbs. of calcium carbonate) per inch of drainage, and this loss may, as time goes on, be expected to influence the changes in the organic matter of the soils.

The average yearly amounts of chlorine per acre in the drainage is very similar to the amount found in the rain, but wide differences occur in some individual years. In the twenty four years during which the chlorine has been determined, the soils of the 20-inch and 40-inch gauges have received from the rain 7·0 and 14·4 lbs. of chlorine per acre in excess of the amounts lost in drainage. The 40-inch gauge has lost 17·5 lbs. of chlorine.

63. "*Benzylidenecamphoroxime.*" By M. O. Forster.

An examination of α aminocamphoroxime, described by Lapworth and Harvey (*Proc. Chem. Soc.*, xviii., 70), appears to have been undertaken with the object of investigating changes which substituted camphoroximes undergo in virtue of the presence of the oximido-group. This has been the author's aim in his study of camphoroxime, which has extended over several years, and contributions to the chemistry of β-chlorocamphoroxime, β-bromocamphoroxime, and α-benzylidenecamphoroxime have been already made. As Lapworth and Harvey have notified privately their intention to abandon the investigation, the author wishes to intimate that he proposes to continue the study of substituted camphoroximes. The following observations relating to benzylidenecamphoroxime are now put on record.

Benzylidenecamphoroxime, $C_{17}H_{21}ON$, dissolves sparingly in boiling alcohol, and crystallises in transparent hexagonal prisms, melting at 197°; for a 2 per cent solution in chloroform, $[\alpha]_D = +389°$. The *benzoyl* derivative crystallises from petroleum in lustrous, well-formed prisms, and melts at 106—107°; for a 2 per cent solution in chloroform, $[\alpha]_D = +246°$. The *phenylcarbamate* separates from alcohol in tufts of long, silky needles, and melts at 169°, when it evolves gas; a 2 per cent solution in chloroform has $[\alpha]_D = +275°$. Nitrous acid converts benzylidenecamphoroxime into the *compound* $C_{17}H_{20}O_2N_2$, which melts at 117°, and gives Liebermann's reaction.

Attempts to convert the oxime into benzylidenecampholenonitrile by the agency of boiling sulphuric acid (25 per cent) have been unsuccessful, but acetyl chloride gives rise to a *nitrile* which is unsaturated and feebly lævorotatory.

The oxime is indifferent towards sodium amalgam, which reduces benzylidenecamphor to benzylcamphor, but sodium in boiling amyl alcohol reduces it to bases which probably represent the α-benzyl derivatives of bornylamine and neobornylamine; the *platinichloride* from the dextrorotatory hydrochloride melts at 235°, whilst the *platinichloride* from the lævorotatory hydrochloride melts at 247°.

ROYAL INSTITUTION.

Annual Meeting, May 1st, 1902.

Sir James Crichton-Browne, M.D., F.R.S., Treasurer and Vice-President, in the Chair.

The Annual Report of the Committee of Visitors for the year 1901, testifying to the continued prosperity and efficient management of the Institution, was read and adopted, and the Report on the Davy-Faraday Research Laboratory of the Royal Institution, which accompanied it, was also read.

Forty-four new Members were elected in 1901. Sixty-three Lectures and seventeen Evening Discourses were delivered in 1901. The Books and Pamphlets presented in 1901 amounted to about 253 volumes, making, with 722 volumes (including Periodicals bound) purchased by the Managers, a total of 975 volumes added to the Library in the year.

Thanks were voted to the President, Treasurer, and the Honorary Secretary, to the Committees of Managers and Visitors, and to the Professors, for their valuable services to the Institution during the past year.

The following gentlemen were unanimously elected as Officers for the ensuing year:—

President—The Duke of Northumberland.

Treasurer—Sir James Crichton-Browne.

Secretary—Sir William Crookes.

Managers—The Right Hon. Lord Alverstone, Sir James Blyth, Bart., Sir Frederick Bramwell, Bart., Dr. Thomas Buzzard, Dr. Donald Hood, Sir Francis Laking, Mr. George Matthey, Dr. Ludwig Mond, Dr. Hugo Muller, Mr. Edward Pollock, Sir Owen Roberts, Sir Felix Semon, The Right Hon. Sir James Stirling, Mr. John I. Thornycroft, and Mr. James Wimshurst.

Visitors—Dr. Henry E. Armstrong, Dr. Charles Edward Beevor, Mr. John B. Broun-Morison, Mr. Francis Elgar, Mr. Francis Gaskell, Dr. Dundas Grant, Lord Greenock, Mr. Maures Horner, Sir Henry Irving, Mr. Wilson Noble, Mr. W. R. Pidgeon, Mr. Arthur Rigg, Mr. W. S. Squire, Mr. Harold Swithinbank, and Mr. Charles Wightman.

NOTICES OF BOOKS.

Lighting by Acetylene; a Treatise for the Practical Lighting Engineer. Containing Elementary Information and Details for those about to take up the Work. By Frederick Dye, M.R.I. London: E. and F. N. Spon, Ltd. New York: Spon and Chamberlain. 1902. Pp. 188.

The purpose of this treatise is, firstly, to make clear what is good and bad in acetylene, or, in other words, to show how its good qualities can be obtained and bad ones avoided or not created; secondly, to be a practical guide to the engineer or fitter undertaking this work.

That Mr. Dye is an optimist as regards acetylene lighting is very evident from a perusal of this book, but we think he goes too far when he states:—"Acetylene can now be made (given a proper generator) by a lad or any unskilled person, without supervision, and with no risk"; and again, speaking of the generator, "it will need no more attention than can be given by a lad, farm-hand, or female servant; and the attendance need not exceed from five to ten minutes per day for a fair-sized plant." Now, a "farm-hand" is generally taken as representing a type of crass stupidity, and the idea of a "fair sized plant" being left to the tender mercies of such a man "without supervision" is at least startling. We may indeed ask, if the above statements are in any way true, why has the author taken the trouble to write this book as a practical guide to engineers and fitters? Surely they are as sharp as the average "lad, farm-hand, or female servant."

After an introduction giving an elementary description of the process of acetylene gas production, the author goes through the history of calcium carbide from which the gas is made. The properties of this gas, with its advantages, risks, &c., are fully described in Chapter II. The next two chapters are devoted to acetylene generators, with types and examples.

The principle of generating acetylene gas is one of the simplest known, yet it has resulted in a vast variety of generating apparatus, usually followed by their appropriate explosions. There are practically four methods now in use; first, that in which the carbide is dropped into water; second, that in which the carbide is placed in trays and the water allowed to drip on it as required;

third, that in which the carbide is put in a holder and dipped into the water, or has the water approach it for a few seconds, whenever the store of gas gets low; and fourth, that in which the carbide is placed in trays and the water allowed to flow to it (not on it), and in about sufficient volume for the quantity of carbide to be attacked. There are modifications of all these methods.

After production, acetylene requires purification, and too much importance cannot be attached to the bearing that generators have on the purity of the gas they produce. At one time the carbide bore all the blame, but now the case is different; experience has shown that the measures to be adopted towards securing pure gas are simply cool generation, and making the gas pass through water as fast as it is made. There are not many different means of purifying acetylene chemically at present, but those that are known are described in Chapter V.

Chapter VI. deals with burners and appliances. The perfect burner has yet to be designed; there are burners, of which the "Naphey" is a well known one, which consume the gas perfectly, but they all become choked with soot at some time. The chief detail of the Naphey burner, and the large number of others made on that principle, is the provision of air-holes leading from the exterior of the nipple to the mixing chamber, or hollow in the centre of the extreme tip; as the gas issues it draws in air which mixes with it, the result being a whiter flame, more perfect combustion, and freedom from smoke.

Chapter VII., and last, is on the legal and other regulations governing the storage of carbide and the fixing of acetylene generating apparatus.

A copy of a licence to keep or store calcium carbide, such as is issued by a country District Council, is given in full, and this represents the documents which lighting engineers are most likely to see, or have to work to, it is recommended that it be carefully read and its contents committed to memory as far as possible.

The book is clearly written and well illustrated, and will doubtless be of value to those connected with the subject of lighting by means of acetylene gas; it also appears to be efficiently indexed.

The Factory and Workshop Act, 1901. Its General Effect and Parliamentary History, with Notes and other Information (including the Full Text of the Act) for the Guidance of Employers of Labour and Others. By C. Willoughby Williams, B.A., and Charles E. Musgrave. London: Effingham Wilson; and A. Stuart, Carthusian Press. 1902. Pp. 196.

The Factory and Workshop Act of 1901 is a measure of considerable importance, and is an advance on previous legislation of a similar kind; it confirms and consolidates the bulk of the old law, and, at the same time, largely extends the State regulation of industry as regards health, sanitation, safety, hours of labour, and other matters. It also throws additional responsibilities upon the Home Office and the local sanitary authorities, and increases the statutory obligations of manufacturers in many material respects.

As the Act only came into operation on January 1st, 1902, any guide to it must be necessarily incomplete, and the authors do not claim to have done more than show, in simple language, its effect as a whole, the principal changes that have been made in the law, and the manner in which the measure passed through Parliament.

The text of the Act is reproduced in full, and the new or modified portions are, as far as practicable, printed in italics; this feature should be extremely useful.

The book is divided principally into three parts; Part I., on the Effect of the Act; Part II., on the Changes in the Law; and Part III. on the Parliamentary History of the Act; then follows the Act with notes and comments, the appendix, containing orders and special rules, and finally the index.

The importance of the interests involved, are shown by the report of the Chief Inspector of Factories and Workshops for the year 1900, in which it is stated that the number of factories on the registers was 95,664, and of workshops 137,648, all of which are affected by this change in the law.

E. Merck, Darmstadt. Annual Report on the Year 1901. Published March, 1902. Pp. 207.

The annual report on the year 1901 follows in form that of previous years. At the commencement there is an exhaustive and detailed article giving the results of original investigations on opium tests.

The succeeding pages contain the usual epitome of the publications of many investigators, and take note of a large number of the recent materia medica. Among the most important items may be mentioned the cacodylates, which are attracting considerable attention at present.

Other preparations dealt with are aconitine, eumenol, tannoform, amyl salicylate, calcium glycerino-arsenate, and many more.

CORRESPONDENCE.

ARSENITE OF SILVER.

To the Editor of the Chemical News.

Sir,—Let me congratulate Mr. Wanklyn on his ability to perform an approximately accurate analysis of silver arsenite in the year 1902; but also let me protest against the libel on Charles L. Bloxam's analytical skill contained in Mr. Wanklyn's words "the drop in percentage of As_2O_3 from 25·88 to 22·19 might have been due to deposit of saltpetre or nitrate of silver in the precipitate!"—I am, &c.

A. G. Bloxam.

Birkbeck Bank Chambers, W.C.,
May 5, 1902.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxiv., No. 15, April 14, 1902.

Researches on Electromotive Forces.—M. Berthelot.—The author performs five series of experiments, from which he draws the conclusion that the electromotive forces, often considerable, which are developed by the union of a great number of elements in a pile, consist of simple actions of neutralisation or analogous reactions, under conditions which are described. These are often insufficient to produce appreciable phenomena of electrolysis, but are especially susceptible of intervening in physiological chemistry. The author also succeeds in making oxidising and reducing reactions take place in saline solutions of piles, and under conditions which he will further investigate, and which are evidently comparable in certain aspects to the reactions which take place during physiological phenomena.

Composition of Gaseous Hydrates.—M. de Forcrand.—In spite of the large number of researches on the subject, the composition of the hydrates of gases appears to be very difficult to determine directly. The author believes that the relation which he gives, $\frac{Q}{T} = 30$, in which Q is the heat of formation of the solidified hydrate and solid water, and T the absolute temperature for which the hydrate has a tension of 760 m.m., allows of a very simple solution of the problem in a great number of cases,

Action of Hydrogen on Strontium Amalgam.—M. Guntz.—In a preceding paper the author shows that whilst heating strontium amalgam in a current of hydrogen, pure strontium hydride, SrH_2, is obtained. He now further investigates the conditions of this reaction, and the precautions which must be taken to obtain the hydride free from mercury. A knowledge of the dissociation tensions of strontium hydride enables the exact conditions of preparation of metallic strontium from this compound to be found.

Compounds of Aluminium with Chromium Sesquioxide.—M. Duboin.—M. Lecoq de Boisbaudran established the conditions under which aluminium combines with chromium oxide, and concluded from his experiments that in rubies the chromium is present in the state of sesquioxide. The author now describes a means of separating the non-combined chromium oxide in these experiments. The oxide, if anhydrous, when heated for one and a-half hours at a white heat, rapidly and totally disappears in chlorate of potash, leaving alumina. By this means the proportion of chromium oxide which is combined with the alumina can be found.

Products of Decomposition of Amidotariric Acid.—M. Arnaud.—By heating amidotariric acid with fuming hydrochloric acid in sealed tubes at 170°, four products of decomposition are obtained. The separation of these bodies is very complicated: they are, on the one hand, undecylamine, $C_{11}H_{23}NH_2$, and pimelic acid, $C_7H_{12}O_4$; and, on the other hand, lauric acid, $C_{12}H_{24}O_2$, and ϵ-amidocaproic acid, $C_6H_{13}NO_2$. All these bodies were purified and analysed and numbers obtained agreeing with the above formulæ. As a result of this research the author is led to adopt for tariric acid the constitutional formula $CH_3-(CH_2)_{10}-C\equiv C-(CH_2)_4-CO_2H$.

Diacetylbenzoylethane and Acetylmethylphenylfurfurane.—F. March.—In a preceding paper the author describes the preparation of diacetylbenzoylethane or phenacylacetylacetone, to which he attributes the formula $(CH_3-CO)_2=CH-CH_2-CO-C_6H_5$. He now further investigates this compound and its derivatives. Phenacylacetylacetone behaves either as a β-diketone or as a γ-diketone. As the former it combines with hydroxylamine, phenylhydrazine, and semicarbazide, forming the isoxazols and the pyrazols. As a γ-diketone it yields a product of dehydratation by losing a molecule of water, this product being acetylmethylphenylfurfurane, and it combines with ammonia in alcoholic solution, giving rise to a phenyl-2-acetyl-4-methyl-5-pyrrol.

Methoethenylbenzene.—M. Tiffeneau.—The author investigates the benzenic carbides with methoethenylic chain, and compares their properties with those of other compounds corresponding to the lateral chain, C_3H_7.

Oxyisopropylphosphinic Acid.—C. Marie.—Oxyisopropylphosphinic acid is formed when a mixture of hypophosphorous acid and acetone is boiled for some time. It is produced in this case by the slow oxidation of the acid $PO_2H_3C_3H_6O$ by means of the oxygen of the air. In consequence of the presence of small quantities of phosphorus and phosphoric acids in the products of this reaction, whose lead salts precipitate at the same time as that of the acid $PO_3H_3C_3H_6O$, this latter is difficult to purify completely. It has a fusion-point of 170—171° lower than that of the acid prepared by the more complicated process described in detail in this paper.

MISCELLANEOUS.

Royal Institution.—A General Monthly Meeting of the Members of the Royal Institution was held on the 5th inst., The Duke of Northumberland, President, in the Chair. Dr. Alfred Hillier, Mr. Sydney Lupton, Mr. Carl F. von Siemens, and Mrs. C. F. von Siemens were elected Members. The special thanks of the Members were returned to Sir Thomas Sanderson, G.C.B., K.C.M.G., for a donation of £5 5s. to the Fund for the Promotion of Experimental Research at Low Temperatures. The Chairman announced that His Grace the President had nominated the following Vice-Presidents for the ensuing year:—The Right Hon. Lord Alverstone, Sir Frederick Bramwell. Dr. Donald Hood, Mr. George Matthey, Dr. Ludwig Mond, The Right Hon. Sir James Stirling, Sir James Crichton-Browne, and Sir William Crookes.

Molybdic Acid.—P. Klason.—The author has repeated the analysis of ordinary molybdate of ammonium, and determined its molecular weight cryoscopically, and has found that its true formula should be $6NH_3.6MoO_3.5H_2O$; this salt may be regarded as intermediate between the di- and tri-ammonic trimolybdates. A salt described by Rammelsberg is in reality the tri-ammonic trimolybdate, $(MoO_4)_3(NH_4)_3H_3+4H_2O$; as for the di-ammonic trimolybdate, $(MoO_4)_3(NH_4)_2H_4+H_2O$, it is formed by the addition of the calculated quantity of hydrochloric acid to a cooled solution of ordinary molybdate; after twenty-four hours a granular crystalline crust is formed, slightly soluble in the cold, and very soluble in warm water, but with decomposition. A mono-ammonic trimolybdate occurs under similar conditions, with the exception of a different quantity of hydrochloric acid; care must be taken to stir continuously, and to add the hydrochloric acid in a fine stream; before long a felted mass of fine needles is formed of the salt $(MoO_4)_3NH_4.H_5$, having properties similar to those of the preceding salt. Commercial "molybdic acid," answers as nearly as possible to the formula $NH_3.3MoO_3.\frac{1}{2}H_2O$. By proceeding as with the other salts, that is by the addition of the calculated quantity of hydrochloric acid to a cooled solution of ordinary molybdate, then filtering after twelve hours to remove the slight turbidity, there are formed, after a few weeks, beautiful clear crystals of the salt $3NH_3.6MoO_3.5H_2O$, intermediate between the mono- and di-ammonic trimolybdates. Having observed that the phosphomolybdate of ammonium contained $12MoO_3$, the author tried, as in the case of the above-mentioned salts, whether he could not produce a tri-ammonic dodecamolybdate. The addition of an excess of hydrochloric acid to a solution of ordinary molybdate gave a white precipitate formed of very small hexagonal prisms of a tri-ammonic pentadecamolybdate, $3NH_3.15MoO_3.20H_2O$, soluble in boiling water; on evaporating at the ordinary temperature the salt re-crystallises without change, but very slowly. To dissolve this salt, it is necessary to plunge it suddenly into boiling water; otherwise, if we heat up gradually in the water, an altogether insoluble hydrate containing $6H_2O$ is formed; the same product is formed by the desiccation of the salt containing $20H_2O$, even at the ordinary temperature. The warm solution of the preceding salt, treated with sal-ammoniac, gives, on cooling, beautiful brilliant prisms of the tri-ammonic dodecamolybdate, $3NH_3.12MoO_3.12H_2O$. The author is of the opinion that all the molybdates, and without doubt even molybdic acid itself, contain the nucleus Mo_3, and it is for this reason that he has not simplified some of the above formulæ; in fact, we get the chloride Mo_3Cl_6, and not $MoCl_2$.—*Berichte*, vol. xxxiv., p. 153.

MEETINGS FOR THE WEEK.

TUESDAY, 13th.—Royal Institution, 3. "English Kings and Kingship," by Prof. F. York Powell, M.A., LL.D.

WEDNESDAY, 14th.—Society of Arts, 4.30. "Boats and Boat Building in the Malay Peninsula," by H. Warington Smyth.

—— Royal Society Conversazione.

THURSDAY, 15th.—Royal Institution, 3. "Recent Geological Discoveries," by A. Smith Woodward, LL.D., F.R.S.

FRIDAY, 16th.—Royal Institution, 9. "The Nebular Theory," by Sir Robert Ball, M.A., LL.D., D.Sc., F.R.S.

SATURDAY, 17th.—Royal Institution, 3. "Poets and Poetry," by Prof. Walter Raleigh, M.A.

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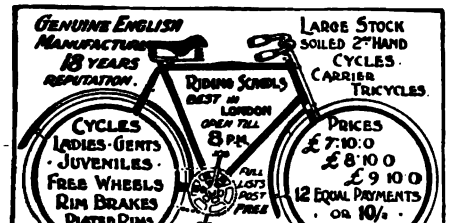

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VOL. LXXXV., No. 2216.

VOLUMETRIC ESTIMATION OF PEROXIDE OF LEAD IN MINIUM.

By M. LIEBIG.

For many years past I have used the following method for the determination of the proportion of peroxide of lead in minium. This method is very convenient, and can be generally recommended on account of the sharpness of the end of the reaction.

Half a gramme of finely powdered minium is placed in a small Erlenmeyer flask, with a little distilled water. By means of a burette 25 c.c. of a decinormal solution of hyposulphite of soda are added, then 10 c.c. of about 30 per cent acetic acid. On well shaking, the mass goes into solution. Then add 10 c.c. of a solution of iodide of potassium at 10 per cent, 2 or 3 c.c. of a solution of iodide of zinc and starch, and titrate the excess of hyposulphite with a decinormal solution of iodine. By multiplying the number of c.c. of iodine used by 239 (239 is the molecular weight of peroxide of lead), we obtain the proportion of peroxide present in the minium.

The end of the reaction is recognised by the change of colour caused by the iodide of lead formed from citron-yellow, as it is at first, to a deep dirty yellow.

As we have said, the reaction is very sharp and very rapid, and it is not necessary to have a very practised eye to perform the operation.—*Zeit. fur Angew. Chem.*, 1901, p. 528.

THE FORMATION AND COMPOSITION OF HYPOCHLORITE OF LIME.

By H. OITZ.

As a result of some experiments on the action of heat, and of dry and moist carbonic acid on several samples of hypochlorite of lime, the author concluded that the action of the chlorine on dry hydrate of calcium should be expressed by the following equation:—

$$2Ca(OH)_2+Cl_2=CaO.Ca(OCl)Cl.H_2O+H_2O,$$

but that the water formed tends to react according to the equation:—

$$CaO.Ca(OCl)Cl.H_2O+H_2O=Ca(OH)_2+Ca(OCl)Cl.H_2O,$$

allowing a fresh quantity of lime to react with the chlorine. The presence of moisture in the lime (due to the water used to slake it) tends to give more importance to the second reaction, and the general result of these successive reactions may be expressed by the equation:—

$$2nCa(OH)_2+(2n-1)Cl_2=$$
$$=(2n-2)Ca(OCl)Cl.H_2O+CaO.Ca(OCl)Cl.H_2O+H_2O.$$

The composition of any sample of chloride should thus be comprised between the formulæ limits $CaO.Ca(OCl).Cl.H_2O$ (35·32 per cent of chlorine) and $Ca(OCl)Cl.H_2O$ (48·96 per cent of chlorine). Neither one nor the other of these formulæ can be accepted as the constitution formula of the compound it represents. To decide on this constitution formula the following facts must be borne in mind:—1. The compound $CaO.Ca(OCl)Cl.H_2O$, stable at 100°, gives off oxygen at a slightly higher temperature, but no chlorine; the residue, $CaO.CaCl_2.H_2O$, only loses its water at a red heat. 2. The other compound, of which the formula is probably $2[Ca(OCl)Cl.H_2O]$, when heated to 100° in a current of dry air, gives off chlorine and water.—*Zeit. fur Angew. Chem.*, vol. xiv., p. 105.

A NEW INVESTIGATION CONCERNING THE ATOMIC WEIGHT OF URANIUM.*

By THEODORE WILLIAM RICHARDS and BENJAMIN SHORES MERIGOLD.

(Continued from p. 224).

The Results of the Analyses of Uranous Bromide.

The method of analysis has been already fully described.

The analyses recorded in the first series were made by adding an excess of silver nitrate to the solution of uranyl bromide. From the ratio of the observed weights of uranous bromide to argentic bromide, the molecular weight of uranous bromide was calculated, that of argentic bromide being assumed to be 187·885. From the results obtained from this preliminary series the weight of silver necessary to precipitate the bromine in 1 grm. of uranous bromide was calculated. In the subsequent determinations the exact weight of silver required was weighed out, as nearly as possible, and dissolved in pure nitric acid with suitable precautions to avoid loss. The exact end-point was reached by standard hundredth normal solutions of argentic nitrate and hydrobromic acid, by means of the nephelometer (Richards, *Proc. Amer. Acad. Arts Sci.*, 1894, xxx., 385; *Zeit. Anorg. Chem.*, 1895, viii., 269). After determining the end-point, a slight excess of argentic nitrate was always added, and the weight of the total argentic bromide determined. Thus from each of these analyses two distinct ratios were obtained as a basis for the calculation of the molecular weight of uranous bromide—the ratio of uranous bromide to argentic bromide, and that of uranous bromide to silver.

As would naturally be expected from the complexity of operations involved, determinations of the sodium in the filtrates from the argentic bromide gave unsatisfactory results. The large quantity of filtrate and wash-waters had to be evaporated to small bulk, the uranium precipitated, and the sodium determined in the residue. It seemed advisable to make a series of separate analyses for sodium only, and use the average percentage of sodium found as a constant correction. This method was used in the work upon cobalt and nickel (*Proc. Amer. Acad. Arts Sci.*, 1899, xxxiv., 339, 365).

Accordingly three alkali determinations were made, *wholly in platinum*, the material not coming in contact with glass at any time except during the original collection and weighing of the sublimed bromide. The sublimate was dissolved in pure water, in a platinum dish, and the uranium was precipitated with pure ammonium sulphide. The ammonium sulphide was freshly prepared for each analysis, wholly in platinum. It left no residue on evaporation in platinum. The precipitated sulphide was digested on the water-bath to expel most of the excess of ammonium sulphide, filtered through a platinum funnel, and the filtrate and wash-water evaporated to small bulk in a platinum dish. The sodium bromide was then converted to sodium sulphate and weighed. The following table contains the data and result:—

No.	Weight of uranous bromide.	Weight sodic sulphate obtained.	Equivalent weight of sodic bromide.	Per cent sodic bromide.
	Grms.	Grm.	Grm.	
1.	1·656	0·00092	0·00133	0·081
2.	2·629	0·00143	0·00207	0·079
3.	1·407	0·00121	0·00175	0·124
			Average	0·095

The average of these three determinations, 0·095 per cent, is practically identical with the amount of sodic bromide found in the cobalt and nickel work, which was 0·10 per cent. The porcelain tubes used in this investigation were of the same manufacture as those used in the

* Contributions from the Chemical Laboratory of Harvard College. From the *Proceedings of the American Academy of Arts and Sciences*, xxxvii., No. 14.

The Atomic Weight of Uranium.

O = 16·000; Ag = 107·93; Br = 79·955.

First Series (*Preliminary*).—UBr_4 : 4AgBr.

No. of analysis.	Total weight of uranous bromide + sodium bromide *in vacuo*.	Weight of uranous bromide corrected for NaBr.	Total weight of silver bromide *in vacuo*.	Weight of silver bromide corrected for NaBr.	Parts of uranous bromide equivalent to 100 parts argentic bromide.	Atomic weight of uranium.
	Grms.	Grms.	Grms.	Grms.	Grms.	
1.	2·20795	2·2058	2·97391	2·9699	74·272	238·36
3.	1·44321	1·4418	1·94272	1·9401	74·316	238·69
5.	1·40639	1·4050	1·89355	1·8910	74·299	238·56
6.	1·17607	1·1749	1·58396	1·5818	74·276	238·39
			Average		74·289	238·50

Second Series.—UBr_4 : 4AgBr.

7.	1·80174	1·7999	2·42588	2·4226	74·296	238·54
8.	1·06723	1·0662	1·43713	1·4352	74·290	238·50
9.	1·85698	1·8551	2·50009	2·4967	74·302	238·59
			Average		74·296	238·54

Third Series.—UBr_4 : 4Ag.

No. of analysis.	Weight of uranous bromide with all corrections.	Weight of silver *in vacuo* (not corrected for sodic bromide).	Weight of silver with all corrections.	Weight of uranous bromide corresponding to 100 grms. silver.	Atomic weight of uranium.
	Grms.	Grms.	Grms.	Grms.	
10 (7).	1·7999	1·39365	1·3918	129·322	238·49
11 (8).	1·0662	0·82559	0·8245	129·315	238·46
12 (9).	1·8551	1·43617	1·4342	129·347	238·60
			Average		238·52

Average of all determinations .. .. 238·52
Average of six final determinations .. .. 238·53

nickel and cobalt work, and since the method of preparation of the three bromides was practically the same, probably the quantity of sodium extracted from the tubes by the action of the hot bromine vapour was the same—*on the average*—in all three cases, and not far from 0·10 per cent. Consequently, in calculating the following results, this value was used as a constant correction. The effect of applying the correction is to raise the calculated atomic weight about two-tenths of a unit. Of course, by this method the quantity of sodic bromide calculated will vary somewhat from the exact quantity present *in individual determinations*. The *average* result, however, will undoubtedly vary but little from the result obtained if the alkali could be determined in each sample. It certainly is very much nearer the truth than the results to be obtained by the cumbersome method of determining the alkali in the filtrate from each precipitation of argentic bromide.

Analysis No. 2 was rejected on account of contamination of the uranous bromide by shreds of asbestos from the packing of the jacket, and No. 4 was not not used because the combustion-tube cracked during sublimation, rendering probable the formation of some oxybromide. The silver required in Analysis No. 6 was determined for practice preparatory to the final series, being 0·9087 grm. when all corrections were applied. It is not included in the table, since its nature was essentially preliminary. As usual, all weighings were reduced to the vacuum standard. While all the weighings were actually made to the hundredths of a milligrm., the final corrected data are rounded off to the nearest tenth of a milligrm., since the deviations of the results show that the hundredths could have had no significance.

The extreme difference between the highest and the lowest values in the preliminary series is 0·33 unit, in the second series 0·09 unit, and in the third series 0·14 unit. At first sight these variations seem large, but their relative magnitude appears smaller when the great molecular weight of uranous bromide, 558·34, is taken into consideration. Thus the extreme percentage error of the preliminary series is 0·06, while those of the last two series are only 0·016 and 0·024 per cent respectively. The so-called "probable error" of the average atomic weight computed from the six analyses numbered 7 to 12 inclusive, if each is given the same weight, is 0·015. That is, according to the theory of least squares, the atomic weight of uranium should be between 238·515 and 238·545.

The magnitude of the maximum deviations in these two final series is, moreover, about as large as whould have been expected from known analytical uncertainty. The observed variation in the amount of sodic bromide, for which a constant correction had to be applied, would account for three-quarters of it, and the rest, corresponding to less than the tenth of a milligrm. in the weighings, might easily be due to unavoidable errors of weighing or manipulation.

Further evidence of the trustworthiness of the figures is to be found in the comparison of the amounts of silver used in Analyses 10, 11, and 12, with the corresponding amounts of argentic bromide, found in Analyses 7, 8, and 9. This comparison is given in the following table, which gives the weights of silver corresponding to 100,000 parts of argentic bromide.

Weight of AgBr *in vacuo*.	Weight of Ag *in vacuo*.	Quotient × 100 = per cent of silver in argentic bromide.
Grms.	Grms.	
2·42588	1·39365	57·449
1·43713	0·82259	57·447
2·50009	1·43617	57·445
	Average	57·447
	Stas found	57·445

The result not only verifies the mechanical work, but affords evidence that the precipitate must have been pure argentic bromide. Clearly, then, the analysis is as accurate as need be. Further repetition of the process might reduce the so-called "probable error," but could not change the average by a significant amount. In the present state of the question, the method seems to have been carried as far as expediency demands.

(To be continued).

CHEMICAL SOCIETIES OF THE NINETEENTH CENTURY.*

By HENRY CARRINGTON BOLTON, Ph.D.

(Continued from p. 221).

FRANCE.

Société industrielle de Mulhouse.

Founded in December, 1825, at Mulhouse (first meeting, May 11, 1826). In 1900: President, Auguste Dollfus; honorary members, 9; resident members, 190; non-resident members, 378; correspondents, 54.

Publication.—Bulletin de la Société industrielle de Mulhausen (*sic*), 1827-1900.

Note.—This is not purely a chemical society, but has a Committee on chemistry, and its Bulletin contains many papers on applied chemistry.

Société chimique de Paris.

Founded June 4, 1857, at Paris. In 1900: President, Edouard Grimaux; members, 365; patrons, 121; life members, 91; corresponding members, 449.

Publications.—(*a*) Bulletin des séances de la Société chimique de Paris, 1858-62; (*b*) Répertoire de chimie pure et appliquée, 1858-63; (*c*) Bulletin de la Société chimique de Paris, 1864-1900; (*d*) Conférence et Leçons, 5 vols.

Association des élèves de M. Fremy.

Founded in 1878 at Paris. In 1900: President, Louis Barthélemy; members, 200.

Publication.—Bulletin trimestriel de l'Association des élèves de M. Fremy, 1878-1900.

Note.—A social organisation, which, however, publishes the work of its members.

Association des Chimistes de sucrerie et de distillerie de France et des Colonies.

Founded in 1883 at Paris. In 1900: President, M. Durin; honorary members, 3; resident members, 160; non-resident members, 710; corresponding members, 395.

Publication.—Bulletin de l'Association des Chimistes de sucrerie et de distillerie de France et des Colonies, 1883-1900.

Association amicale des anciens élèves de l'École de physique et de chimie industrielle de la ville de Paris.

Founded in 1885 at Paris. In 1900: President, Octave Boudouard; honorary members, 41; members, 300.

Publication. — Bulletin mensuel de l'Association amicale des anciens élèves de l'École de physique et de chimie industrielle de la ville de Paris, 1885-1900. Annuaire (&c.), 1885-1900.

Association amicale des anciens élèves de l'École de chimie industrielle de l'Lyon.

Founded in 1886 at the Institut chimique de Lyon. In 1900: President, Alphonse Seyewitz; honorary members, 6; members, 104.

Publication.—Bulletin des séances de l'Association amicale des anciens élèves de l'École de chimie industrielle de Lyon.

Syndicat central des chimistes et essayeurs de France.

Founded in 1890 at Paris. In 1900: President, Ferdinand Jean; members, 125.

Publications.—Revue de chimie analytique appliquée à l'industrie, 1893-98. Annales de chimie analytique appliquée à l'industrie became the organ of the society in 1899; the Annales had been established in 1896, and was united with the Revue (above named) in 1899.

Société chimique du Nord de la France.

Founded at Lille in 1891. In 1900: President, A. Pouriez; members, 100.

Publication.—Bulletin mensuel de la Société chimique du Nord de la France, 1891-1900.

Association amicale des anciens élèves de l'Institut chimique de Nancy.

Founded November 9, 1892, at Nancy. In 1900: President, M. Noel; honorary members, 8; patrons, 7; members, 52; associates, 75.

Publication.—Bulletin (annuel) de l'Association.

Association amicale des élèves et anciens élèves du laboratoire d'Enseignment pratique appliquée de l'université de Paris.

Founded in 1897 at Paris. In 1900: President, M. Loyer; honorary members, 12; members, 110.

Publication.—Gazette de chimie, Paris, 1900.

GERMANY.

Verein für die Rübenzucker Industrie im Zollverein (later, des deutschen Reichs; later, Verein der deutschen Zuckerindustrie).

Founded in 1850 at Berlin. In 1900: President, De Coste; members, 447.

Publication.—Zeitschrift des Vereins (&c.), 1850-1900.

Deutsche chemische Gesellschaft zu Berlin.

Founded in 1867 at Berlin. In 1900: President, G. Volhard; honorary members, 15; life members, 92; members, 2637; associates, 372.

Publication. — Berichte der deutschen chemischen Gesellschaft zu Berlin, 1868-1900. Since 1897 also Chemisches Centralblatt (established in 1830).

Verein analytischer Chemiker.

Founded in 1878 at Magdeburg, and merged in 1887 with the Deutsche Gesellschaft für angewandte Chemie. *See* Verein deutscher Chemiker.

Publication.—Correspondenzblatt des Vereines analytischer Chemiker, 1878-80.

Freie Vereinigung bayerischer Vertreter der angewandten Chemie.

Founded in May, 1883, at Munich. In 1900: President, Albert Hilger; honorary members, 2; members, 124; correspondents, 69.

Publications.—Bericht über die 1 (-18) Versammlung der freien Vereinigung bayerischer Vertreter der angewandten Chemie, 1883-1900. Also reports in Forschungsberichte über Lebensmittel und ihre Beziehung zur Hygiene, 1894-97; and in Zeitschrift für Untersuchung der Nahrungs- und Genuss-Mittel, 1898-1900.

Verein deutscher Berufs-Chemiker.

Founded in 1887 at Dresden.

Publication.—The "Chemiker und Drougist" (Dresden, 1885) had in 1887 the sub-title Correspondenzblatt des Vereins deutscher Berufs Chemiker. This title was dropped in 1888.

Deutsche Gesellschaft für angewandte Chemie.

Founded November, 1887, at Berlin, absorbing the Verein analytischer Chemiker. In 1896 the Society became Verein deutscher Chemiker, *q. v.*

* Read at the Twenty-fifth Anniversary of the American Chemical Society, held in New York City, April 12—13, 1901. From advance proofs of the *Smithsonian Miscellaneous Collections*, 1901.

Publication.—Zeitschrift für angewandte Chemie, 1888-1900. This was begun as Zeitschrift für die chemische Industrie in 1887.

VEREINIGUNG ÖFFENTLICHER ANALYTISCHER CHEMIKER SACHSENS.

Founded in 1890 at Plauen in Vogtland. In 1900: President, Arthur Forster; members, about 25.

Publication.—Zeitschrift für öffentliche Chemie, 1897-1900. Also Bericht über die Hamptversammlung des Vereines öffentlicher analytischer Chemiker Sachsens.

VEREIN AKADEMISCH-GEBILDETE (later, DEUTSCHER) ZUCKERTECHNIKER.

Founded in 1891 at Berlin. In 1900: President, H. Claassen; honorary members, 1; members, 406; correspondents, 3.

Publication.—Zeitschrift des Vereines akademischegebildete Zuckertechniker, 1891-92.

NOTE.—The organ of publication changed several times.

VERBAND DES LABORATORIUMS-VORSTÄNDE VON DEUTSCHEN HOCHSCHULEN.

Founded in 1898 (?).

ZWEIGVEREIN DER ZUCKERTECHNIKER FÜR DAS AUSLAND.

Founded at Berlin. In 1901: President, C. Huck.

DEUTSCHE ELEKTROCHEMISCHE GESELLSCHAFT.

Founded in October, 1894, at Berlin. In 1900: President, J. H. van 't Hoff; members, about 700.

Publication. — Bericht der deutschen elektrochemischen Gesellschaft, 1894-1900.

VEREIN DEUTSCHER CHEMIKER.

Founded in 1896 at Berlin, as successor to Gesellschaft für angewandte Chemie (1887). Its seat is the residence of the President for a given year. In 1900: President, H. Caro; honorary members, 4; members, 2271. Embraced in 1900 the following sections (Bezirk-Vereine):—Aachen, Belgien, Berlin, Frankfurt, Hamburg, Hannover, Mittel- Franken, Mittel- und Niederschlesien Oberrhein, Oberschlesien, Pommern, Rheinland, Rheinland-Westphalen, Saar, Sachsen-Anhalt, Sachsen-Thüringen, Württemberg.

Publication.—Zeitschrift für angewandte Chemie, 1887-1900. *Cf.* Deutsche Gesellschaft für angewandte Chemie.

VERBAND SELBSTÄNDIGER ÖFFENTLICHER CHEMIKER DEUTSCHLANDS.

Founded May 30, 1896, at Nürnberg. In 1900: President, Robert Kayser; members, 161; Associates, 102.

Publication. — Zeitschrift für öffentliche Chemie (established in 1895), 1897-1900. Also Vol. I. as Vol. III., 1897, of Centraiblatt für Nahrungs- und Genussmittel Chemie, sowie Hygiene.

(To be continued).

The Estimation of Chromium and Iron by a Mixture of Iodide and Iodate of Potassium.—A. Stock and C. Masaciu.—The method in question has already been applied by M. Stock (*Berichte*, vol. xxxiii., p. 548) to the estimation of aluminium, and gave better results than the precipitation by ammonia. To the solution of chromium or iron (FeO or Fe_2O_3), slightly acidulated, an excess of a mixture of iodide and iodate of potassium is added, then, after a few minutes, hyposulphite of soda is added to decolorise the iodine set free; a few more cubic centimetres of this salt are then added, and the liquid, with the precipitate in suspension, is boiled for half-an-hour. The precipitate is collected on a filter in a warm funnel, then washed, dried, and weighed.—*Berichte*, vol. xxxiv., p. 467.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, Wednesday, April 30th, 1902.

Prof. EMERSON REYNOLDS, Sc.D., V.P.R.S., President, in the Chair.

CERTIFICATES were read for the first time in favour of Messrs. Percy J. Ferris, Heathfield, Seymour Grove, Old Trafford, Manchester; Alexander Gow, 38, Clarendon Street, Cambridge; Archie Cecil Osborn Hann, 54, Wood Vale, Forest Hill, S.E.; Walter Ernest Harrison, 43, Mostyn Road, Handsworth, Staffs.; Sinnott Valentine O'Connor, 14, Selskar Street, Wexford; Stephen Jamieson Ralph, 22, Stanley Place, Eccleston Square, S.W.; Edwin Ralphs, Hong-Kong.

A ballot for the election of Fellows was held, and the following were declared duly elected:—James Samuel Ashe; Frederic Guy S. Baker, B.A.; Cyril Bergtheil; Richard Blenkinsop; Ernest Boardman, B.Sc.; Alexander Bruce, B.Sc.; Fred. Carrodus; G. H. A. Clowes, Ph.D.; Arthur James Cook; James Codrington Crocker, B.A.; Henry Crookes; Charles Benson Davis; Henry Arthur Dobson; Walter Everitt; J. Arthur Gill; John T. Griffiths; Gilbert Gunn; Arthur Hopwood; Frank Eustace King, B.Sc.; Sidney Isaac King; James Kewley, B.A.; A. Lionel Landau; C. H. Lockitt, B.A.; Alexander Macknight; William H. Martindale, Ph.D.; Robert Mathieson; George A. H. Mence, B.Sc.; John Wicliffe Peck; Walter Ramshaw; Cecil Revis; Jonathan Hugh Roberts, B.A.; William Scholes; Thomas Taylor; Arthur Lea B. Tindall, M.A.; Stephen Herbert Trimen; William Wright Tunnicliffe; William Charles Wain; Edward John Wilkinson; Frank Stanley Wood; Thos. A. Young.

Of the following papers, those marked * were read:—

*64. "*The Preparation of Absolute Alcohol from Strong Spirit.*" By S. YOUNG, D.Sc., F.R.S.

The strongest spirit that can be obtained by distillation with the most perfect still-head contains less than 96 per cent by weight of pure alcohol. In order to remove the remainder of the water, dehydrating agents have always been employed, but they do not give very satisfactory results; according to Mendeleeff, freshly ignited lime is the only one by means of which pure dry alcohol can be prepared, and even with this substance special precautions have to be taken to ensure success. More recently, Squibb has obtained alcohol of still lower specific gravity by the long continued action of lime in a percolator, but his results, unlike those of Mendeleeff, were somewhat variable.

The method of dehydration recommended by the author depends on the fact that when a mixture of alcohol, benzene, and water is distilled, separation tends to take place into (1) a ternary mixture of alcohol, benzene, and water boiling constantly at 64·85°; (2) one of three possible mixtures of constant boiling-point, (*a*) alcohol-benzene, b. p. 68·25°; (*b*) benzene-water, b. p. 69·25; (*c*) alcohol-water, b. p. 78·15°; (3) one of the three pure substances, alcohol, b. p. 78·3°; benzene, b. p. 80·2; water, b. p. 100°.

For example, when a mixture of equal weights of 93 per cent alcohol and benzene is distilled, the fractions are (1) the ternary mixture containing, theoretically, the whole of the water; (2) the binary alcohol-benzene mixture; (3) pure alcohol.

Pure benzene and dilute alcohol are easily recovered from the ternary and binary mixtures, and there is practically no loss of material.

Theoretically, a single distillation should be sufficient to remove the whole of the water, but the separation is a difficult one, and even with a very efficient still-head a second, or even third, distillation with more benzene is necessary; the water may thus be completely eliminated.

but there appears to be a trace of benzene left in the alcohol. This trace of benzene may be completely removed by distillation of the dehydrated alcohol with *n*-hexane; the binary hexane-alcohol mixture which comes over first carries the benzene with it, and no hexane is left in the residual alcohol.

The observed specific gravities of dehydrated alcohol are as follows:—

Mendeleeff, by means of lime	0·80625	at 0°/4°
Young, by means of benzene	0·80634	„
„ „ benzene and hexane	0·80627	„

*65. "*On the Properties of Mixtures of the Lower Alcohols with Water.*" By S. Young, D.Sc., F.R.S., and Miss E. C. Fortey, B.Sc.

Methyl alcohol does not form a mixture of minimum boiling-point with water, and, contrary to general opinion, dry methyl alcohol can be easily obtained by distillation of the dilute alcohol with an efficient still-head. The alcohol so obtained is drier than when the strongest dehydrating agents are used.

As regards the purification of the other six alcohols investigated, distillation with benzene gave the best results with ethyl, *iso*propyl, *n*-propyl, and tertiary butyl alcohols; this treatment was also found useful in the case of *iso*butyl and *iso*amyl alcohols for the removal of lower homologues. All these alcohols were found to form mixtures of minimum boiling-point with water. The boiling-points and the composition of these mixtures of constant boiling-point were determined, also the contraction which occurs on mixing the alcohols with water in various proportions, and the approximate temperature changes for certain mixtures. It was found that (*a*) the differences in boiling-point between the alcohol and the alcohol-water mixtures of constant boiling-point, (*b*) the molecular percentages of water in these mixtures, follow the same order as the boiling-points of the alcohols themselves. So far as experiments have been made, the heat evolution diminishes or the heat absorption increases as the boiling-points of the alcohols rise.

As regards the primary alcohols, the percentage contractions on mixing with water diminish as the boiling-points of the alcohols rise, but irregularities were observed with the secondary and the tertiary alcohols investigated.

The experiments show that as the molecular weights of the alcohols increase, the attractions of their molecules for those of water diminish, but the constitution as well as the composition of the alcohols must be taken into account. The constant boiling-point mixtures which the alcohols form with water are in no case hydrates of the alcohols.

*66. "*On the Properties of Mixtures of the Lower Alcohols with Benzene and with Benzene and Water.*" By S. Young, D.Sc., F.R.S., and Miss E. C. Fortey, B.Sc.

Of the seven alcohols examined, all except *iso*amyl alcohol form mixtures of constant boiling-point with benzene. The boiling-points of these mixtures and the molecular percentages of benzene in them follow the same order as the boiling-points of the pure alcohols. The boiling-point of the *iso*butyl alcohol-benzene mixture is very near that of benzene itself; neither *iso*amyl alcohol nor any other alcohol with a higher boiling-point can form a mixture of constant boiling-point with benzene.

Of these alcohols, only ethyl, *iso*propyl, *n*-propyl, and tertiary butyl alcohols form ternary mixtures of constant boiling-point with benzene and water. When a mixture of methyl alcohol, benzene, and water is distilled, the first fraction consists of the binary alcohol-benzene mixture, and when a mixture of either *iso*butyl or *iso*amyl alcohol with benzene and water is distilled, the first fraction consists of the binary benzene-water mixture. No alcohol of this series with a boiling-point higher than that of *iso*butyl alcohol can form a ternary alcohol-benzene-water mixture of minimum boiling-point; with these higher alcohols, the first fraction will always consist of the binary benzene-water mixture.

The boiling-points and the composition of the binary alcohol-benzene and the ternary alcohol-benzene-water mixtures have been determined.

*67. "*Fractional Distillation as a Method of Quantitative Analysis.*" By S. Young, D.Sc., F.R.S., and Miss E. C. Fortey, B.Sc.

It has already been pointed out (Young, *Journ. Soc. Chem. Ind.*, 1900, xix., 1072) that the composition of a mixture of two homologous substances can be ascertained with a fair degree of accuracy from the results of one or more fractional distillations with a very efficient still-head without bringing about anything like a complete separation of the components.

The method has now been found applicable, not only to mixtures which separate normally into their original components, whether homologous or not, but also to cases in which binary or ternary mixtures of constant boiling-point are formed, such mixtures behaving, as regards distillation, like pure substances. It depends on the following facts:—In the case of a mixture which tends to separate into two components, the weight of distillate that comes over below the temperature midway between the boiling-points of the two components—whether single substances or mixtures of constant boiling-point—is almost exactly equal to that of the more volatile component. If the original mixture tends to separate into more than two components, the weight of these components will be very nearly equal to the weight of distillate below the first middle point, the weights of distillates between the successive middle points, and the weight above the last middle point respectively.

When a binary mixture of constant boiling-point is formed, the method may be employed for the determination of its composition if that of the original mixture is known, or of the original mixture if that of the binary mixture is known; when the original mixture tends to separate into a ternary mixture, a binary mixture, and a single substance, the composition of the ternary mixture may be ascertained, if that of the binary mixture and of the original mixture are known.

There are a few cases such as that of ethyl alcohol and water, or of benzene and *n*-hexane, to which the method is not applicable. In these, the curve representing the relation between boiling-point and molecular composition is exceedingly flat where the more volatile component is in large excess. When this is so, the separation by fractional distillation of the more volatile component, whether a single substance or a mixture of constant boiling-point, is practically impossible.

*68. "*On the Vapour Pressures and Boiling-points of mixed Liquids.*" By S. Young, D.Sc., F.R.S.

The question as to what should be regarded as the normal behaviour, with reference to their vapour pressures and boiling-points, of mixtures of two liquids soluble in each other in all proportions, has been discussed by several investigators.

It seemed probable that the simplest possible relations would be observed in the case of two such closely related substances as chlorobenzene and bromobenzene, the critical points of which are the same, whilst the ratio of the boiling-points on the absolute scale is constant at all equal pressures. Other simple physical relations exist between these substances, and no perceptible heat change or volume change occurs on mixing them.

The boiling-points of several mixtures of chlorobenzene and bromobenzene were determined over a range of about 100 m.m., and the results are in accurate accordance with the law which Van der Waals states should gold good for two liquids the critical points of which are equal, and for which $a_{1\cdot2} = \sqrt{a_1 . a_2}$ (Galitzine, Berthelot), where $a_{1\cdot2}$ represents the mutual attraction of the unlike molecules and a_1 and a_2 the attractions of the like molecules.

The law may be stated thus :—The relation between the vapour pressures and the molecular composition of mixtures of such substances is represented by a straight line; or $P = \frac{p.P_A + (100-p)P_B}{100}$, where P, P_A, and P_B are the vapour pressures of the mixture, and of the two pure components, A and B, at the same temperature, and p is the molecular percentage of one of them.

*69. "*The Correction of the Boiling-points of Liquids from Observed to Normal Pressure.*" By S. Young, D.Sc., F.R.S.

Crafts (*Ber.*, 1887, xx., 709) gave a convenient table for the correction of the boiling-points of liquids from observed to normal pressure. There are, however, a few misprints in the table, and since then accurate determinations of the vapour pressures of a large number of pure substances have been made; it therefore seems desirable to give a revised and enlarged table for reference.

Crafts gave mean values of $\frac{\Delta t}{\Delta p} \cdot \frac{1}{T}$ for pressures between 720 and 770 m.m., but it seems more convenient, on the whole, to give the values of $\frac{dt}{dp} \cdot \frac{1}{T} = C$ at 760 m.m.

Various relations exist between the values of C and the chemical composition and constitution of the substances considered, and there is clearly a connection between the variations in the values of C and of other constants which have been considered in previous papers, such as $\frac{De}{D'c}$, the ratio of the actual to the theoretical density at the critical point.

*70. "*Vapour Pressures and Specific Volumes of* Iso*propyl* Iso*butyrate.*" By S. Young, D.Sc., F.R.S., and Miss E. C. Fortey, B.Sc.

By the electrolysis of potassium *iso*butyrate, the yield of di*iso*propyl was found to be very poor, but a fair quantity of *iso*propyl *iso*butyrate was obtained. As this ester contains two *iso*-groups, it was thought that it would be of interest to determine its vapour pressures, specific volumes, and critical constants. For the sake of comparison, a second specimen of the ester was prepared from *iso*propyl alcohol and *iso*butyric acid, and the boiling-points and specific gravities of the two specimens were found to agree perfectly.

Unfortunately, *iso*propyl *iso*butyrate is much less stable than any of the ten esters previously examined (Young and Thomas, *Trans.*, 1893, lxiii., 1191), for some decomposition takes place at 230°, and the rate of decomposition increases rapidly with rise of temperature. It was therefore impossible to make trustworthy determinations of the critical temperature or pressure, so only the vapour pressures and specific volumes at and below 230° are given.

Discussion.

Professor Thorpe said, in reference to the observations which had been made by the author concerning the properties of the hydrated alcohols, and the thermal disturbance and alteration in volume accompanying the admixture of alcohols with water, that it appeared to be a tradition in the Revenue Service that on mixing ordinary (ethyl) alcohol and water, the combination, or association, of water and alcohol was only completed after a certain lapse of time, or, in other words, the mixture required to stand for some time before it acquired a constant volume, or, what comes to the same thing, a constant relative density. On what experimental evidence this belief was based was unknown to him, and direct observation made under his direction in the Government Laboratory, by Mr. John Holmes, showed that in reality the tradition had no foundation in fact. It probably had its origin in a statement in the original paper by Blagden and Gilpin that the mixtures of alcohol and water employed by them in their classical experiments, when made, were set aside for some months, in order that, as stated by them, the "interpenetration" of the parts should be complete. It seems to have been taken for granted, in fact, that interpenetration, as something apart from complete and uniform miscibility, occurred on admixture, and that this operation required a certain and considerable interval of time.

The observation on the solubility of copper sulphate in methyl alcohol had been frequently made, and he believed some reference was made to it in a paper by the late Mr. Rodger and himself (*Phil. Trans.*, 1894, clxxxv., 530). According to Forcrand, the bluish compound has the composition $CuSO_4,2CH_3OH$.

Some years ago, he made the observation that a mixture of carbon tetrachloride and methyl alcohol could be obtained which boiled several degrees lower than the boiling-point of the more volatile constituent. He had studied the relation between the boiling-point and composition of the mixture, determining the composition from the vapour density of the fractions (*Trans.*, 1879, xxxv., 544). He would be interested to know how far the quantitative results he had obtained were in conformity with the relations indicated by Dr. Young in the case of other mixtures of miscible liquids.

Mr. Pickering said that although the existence of a constant boiling-point mixture of two substances cannot be taken as proof that these two substances are combined, yet the fact that the composition is affected to a certain extent by pressure is no proof of the contrary. Aqueous solutions of hydrochloric acid can be distilled unchanged when the composition is approximately $HCl,8H_2O$, and Roscoe and Dittmar were held to have disproved the existence of any such hydrate by showing that the composition is affected by pressure; yet, Berthelot proved that this variability did not by any means negative the existence of a hydrate, the mixture in question being one of comparative, though not of absolute, constancy. Indeed, we could not expect the composition to be unaffected by pressure, unless the compound were perfectly stable, and there were no products of its dissociation present.

The fact, also, that water can be crystallised from a solution does not in any way disprove the existence of a hydrate in solution. The water may be present only as one of the dissociation products of the hydrate, and whether it or the hydrate separates out depends on the circumstances of the crystallisation. Numerous instances in point might be given: thus, water may be crystallised from a 20 per cent solution of sodium hydroxide, though such a solution will, under other conditions, yield crystals of the heptahydrate, of which it is chiefly composed.

Dr. Divers mentioned, in connection with the author's account of the solution of anhydrous copper sulphate, with blue colour, in anhydrous methyl alcohol, that W. Wislicenus and Stoeber had recently pointed out (*Ber.*, 1902, xxxv., 540) that anhydrous cupric acetate dissolves in methyl alcohol when boiled with it, giving a deep green solution which deposits crystalline cupric methoxide-acetate, $CH_3OCuC_2H_3O_2$, if the boiling be prolonged.

Professor Young replied that they had not yet investigated the mixture of carbon tetrachloride and methyl alcohol, but agree that it would be interesting to determine the composition of the mixture of minimum boiling-point by the distillation method, in order to compare the result with that obtained by Professor Thorpe from the vapour density.

He did not regard the variability of the composition of the mixtures of minimum boiling-point with the pressure as a sufficient proof, but only as strong confirmatory evidence as to the non-existence of hydrates of the alcohols. The reasons for believing that such hydrates do not exist are fairly numerous, whilst the only ground for assuming that the liquids under consideration are hydrates seems to be that they boil at a constant temperature. Yet the very fact that the boiling-points are lower than those of the alcohols themselves or than that of water indicates that the attraction of the unlike molecules is small compared with the attractions of the

like molecules, and this could hardly be the case if hydrates were formed. With regard to the interesting action of anhydrous copper acetate on methyl alcohol, he might mention that Beilstein describes a crystalline alcoholate of copper sulphate, $CuSO_4.CH_3OH$.

*71. "*The Preparation of Highly Substituted Nitroaminobenzenes.*" By K. J. P. Orton.

The methods at present known of preparing nitroaminobenzenes from anilines are scarcely applicable when the anilines possess two ortho-substituents. The following procedure has been devised, and in most cases gives a quantitative conversion of the aniline into the nitroamine.

To a solution of the aniline (1 part) in glacial acetic acid (25 parts) is added nitric acid (sp. gr. 1·5; 0·75 to 1 part); acetic anhydride (0·75 to 1 part) is slowly added to the cooled mixture. Heat is developed, and the aniline nitrate dissolves, forming a solution of the nitroamine.

By this means, the following nitroamines have been prepared:—

1-Nitroamino-3-tribromobenzene and 1-nitroamino-*s*-trichlorobenzene (*Proc.*, xviii., 58); 1-*nitroamino*-2 : 6-*dichloro*-4-*bromobenzene*, and 1-*nitroamino*-2 : 4-*dichloro*-6-*bromobenzene*, $C_6H_2Cl_2Br{\cdot}NHNO_2$, 1-*nitroamino*-2-*chloro*-4 : 6-*dibromobenzene*, and 1-*nitroamino*-4-*chloro*-2 : 6-*dibromobenzene*, $C_6H_2ClBr_2{\cdot}NHNO_2$; these four substances crystallise in needles (from water), and all melt and decompose at 136—138°; their *barium* salts crystallise in plates with $1H_2O$. The N-*methyl ether* of nitroamino-*s*-tribromobenzene, $C_6H_2Br_3{\cdot}NMeNO_2$, obtained from the sodium salt and methyl iodide, forms four-sided prisms melting at 95·5°.

1-*Nitroamino*-2 : 4-*dibromo*-6-*nitrobenzene*,—

$$NO_2{\cdot}C_6H_2Br_2{\cdot}NHNO_2,$$

m. p. 91—92°, forms yellow plates (from water); 1-*nitroamino*-2 : 3 : 4 : 6-*tetrabromobenzene*, plates melting and decomposing at 136°; 1 *nitroamino*-2 : 4 : 6-*tribromo*-3-*nitrobenzene*, yellow four-sided prisms, melting and decomposing at 108—109°; 1-*nitroamino*-2 : 4-*dichlorobenzene*, lustrous leaflets from petroleum, melting at 55—56°. The *barium* salt of the last-mentioned substance crystallises in plates with $3\frac{1}{2}H_2O$.

4-*Nitroamino*-3 : 5-*dibromotoluene*,—

$$CH_3{\cdot}C_6H_2Br_2{\cdot}NHNO_2,$$

forms slender needles melting and decomposing at 122—123°; 2-*nitroamino*-3 : 5-*dibromotoluene*, needles, melting at 112°, and decomposing at 122°; the *barium* salts from each substance crystallise with $1H_2O$.

When nitroamino-*s*-trisubstituted benzenes contain a bromine atom in the para-position relative to the $\cdot NHNO_2$ group, the nitro-group is capable of displacing the *p*-bromine atom, giving a di-*o*-substituted *p*-nitroaniline. The majority of these nitroamines dissolve in concentrated sulphuric acid, forming a deep purple or violet solution; in the case of nitroamino-2 : 4-dichlorobenzene, which has an *o*-hydrogen atom, addition of water immediately destroys the purple colour, and a yellow precipitate of 2 : 4-dichloro-6-nitroaniline is formed; in the case of the other nitroamines, in which the hydrogen atoms in positions 2, 4, and 6 are replaced by other atoms or groups, water precipitates from the purple solution a red solid; the latter contains among other substances a di-*o*-substituted *p*-nitroaniline if the original nitroamine had a *p*-bromine atom. The purple solutions in sulphuric acid may possibly correspond to an intermediate stage between the nitroamine and the nitrated aniline.

*72. "*The Atomic Weight of Tellurium.*" Preliminary notice. By A. Scott.

The many attempts to determine the atomic weight of tellurium which have been published since Mendeleeff published his well-known table of elements, indicate very clearly the general recognition that experimental results did not sanction the assignment of a lower atomic weight to tellurium than that of iodine. The problem was rendered more complicated by the variations in the numbers obtained by the same experimenter when he employed different methods in his determinations. This gave rise to the suspicion that the substance purified by the best methods was, or at least might be, a mixture of the true tellurium with a higher homologue. All the most recent researches, and especially those of Pellini (*Ber.*, 1901, xxxiv., 3807) and of Köthner (*Annalen*, 1901, cccix., 1), seem conclusively to prove, however, that the atomic weight of tellurium is very near 127·64, that of iodine being 126·85. For more than a year the author has been at work on this subject, and his results are entirely in agreement with those of the above-mentioned experimenters. It was obvious that the most satisfactory substance for such determinations should contain both tellurium and iodine, and be weighted with as little as possible of other elements. Such a substance is trimethyl tellurium iodide, which is easily prepared and purified, is very stable, and non-hygroscopic. When tellurium is heated to 80—100° in a sealed tube with methyl iodide (Demarçay, *Bull. Soc. Chim.*, 1883, xl., 100), dimethyl tellurium di-iodide is readily formed; when this is treated with reducing agents in presence of methyl iodide, trimethyl tellurium iodide, $Te(CH_3)_3I$, is readily obtained. The corresponding bromide is a salt very well adapted for titration with silver by the method of Stas. Preliminary determinations so far agree with all the best results hitherto published in proving that the atomic weight of tellurium is nearly a unit higher than that of iodine.

In the following determinations, all the weights given are corrected for errors in face values of the weights, as well as being brought to vacuum standards:—

$Te(CH_3)_3I$.	AgI.	
1·7461 gave	1·3688	∴ $Te(CH_3)_3I = 299·62$ and $Te = 127·70$.
6·6425 ,,	5·20575	∴ $Te(CH_3)_3I = 299·58$,, $Te = 127·66$.
8·0628 ,,	6·3181	∴ $Te(CH_3)_3I = 299·61$,, $Te = 127·69$.

By titration:—

2·4294 $Te(CH_3)_3Br$ required 1·0373 Ag ∴ $Te(CH_3)_3Br = 252·79$ and $Te = 127·77$.

6·8424 $Te(CH_3)_3Br$ required 2·9201 Ag ∴ $Te(CH_3)_3Br = 252·90$ and $Te = 127·88$.

The second sample of bromide was not so pure as the first, as it was the second crop of crystals obtained from the mother-liquors. The mean value is therefore $Te = 127·74$ taking all five determinations, or 127·70 if only the first four be taken.

Pellini gives 127·64.
Köthner ,, 127·63.

The author indicated by a graphic representation of the quotients—

$$\frac{F}{O} = 1·190, \frac{Cl}{S} = 1·105, \frac{Br}{Se} = 1·009, \frac{I}{Te} = 0·993,$$

obtained by dividing the atomic weights of the halogen elements by those of the corresponding members of the oxygen group respectively, why he thought it very unlikely that the atomic weight of iodine should exceed that of tellurium.

In making the necessary calculations, the following values were taken:—C = 12·00; H = 1·0075; I = 126·85; Br = 79·95 (O = 16).

Two syntheses of silver iodide were made, as follows:—

Ag.	AgI.	
4·6240 gave	10·0634	∴ AgI = 234·89 and I = 126·96
6·39978 ,,	13·92913	∴ AgI = 234·91 ,, I = 126·98

Substituting I = 126·97 for I = 126·85 in the first three calculations increases the values for $Te(CH_3)_3I$ by 0·15, and those for Te by 0·03.

73. "*Nitrogen Bromides containing the Propionyl Group.*" By F. D. Chattaway.

Propionanilide and the bromopropionanilides readily react with hypobromous acid, the imino-hydrogen being replaced by bromine, a hypobromite being probably formed as an intermediate product.

The action is a reversible one, and when nitrogen

bromides are placed in water the opposite change takes place until a position of equilibrium is reached.

Except in colour, these nitrogen bromides closely resemble the corresponding nitrogen chlorides. All show the characteristic behaviour of the nitrogen halogen linkage, and readily react with hydriodic acid, sulphurous acid, hydrogen sulphide, alcohol, and potassium cyanide.

When an unsubstituted or partially substituted phenyl residue is attached to the nitrogen, they very readily undergo isomeric change.

Interchange between bromine attached to the nitrogen and hydrogen in the ortho-position in an unsubstituted nucleus has not been observed. Apparently the halogen cannot pass from the nitrogen to a meta-position.

All nitrogen bromides, when treated with an excess of hydrobromic acid, are decomposed, bromine being liberated and the corresponding anilide re-formed. On the other hand, when an anilide is treated with an excess of bromine, a nitrogen bromide and hydrobromic acid are produced.

A large excess of hydrobromic acid or the continuous removal of the free bromine causes the anilide to be re-formed, whilst the addition of a salt of a weaker acid which can remove the hydrobromic acid causes the action to proceed in the opposite direction, and a nitrogen bromide is produced. The reversible nature of this reaction is most clearly seen when the nucleus is already fully substituted, as otherwise isomeric change and substitution into the ring may take place.

The following substances have been prepared :—

Propionylphenyl nitrogen bromide, $C_6H_5{\cdot}NBr{\cdot}CO{\cdot}C_2H_5$, pale yellow pyramids, m. p. 88°; *p-bromopropionanilide*, $C_6H_4Br{\cdot}NH{\cdot}CO{\cdot}C_2H_5$, rectangular plates, m. p. 149°; *propionyl-p-bromophenyl nitrogen bromide*, $C_6H_4Br{\cdot}NBr{\cdot}CO{\cdot}C_2H_5$, yellow prisms, m. p. 78°; *propionyl-p-bromophenyl nitrogen chloride*, $C_6H_4Br{\cdot}NCl{\cdot}CO{\cdot}C_2H_5$, colourless plates, m. p. 59°; *o-bromopropionanilide*, $C_6H_4Br{\cdot}NH{\cdot}CO{\cdot}C_2H_5$, colourless prisms, m. p. 93°; *propionyl-o-bromophenyl nitrogen bromide*,—

$$C_6H_4Br{\cdot}NBr{\cdot}CO{\cdot}C_2H_5,$$

yellow prisms, m. p. 117° ; *propionyl-o-bromophenyl nitrogen chloride*, $C_6H_4Br{\cdot}NCl{\cdot}CO{\cdot}C_2H_5$, colourless rhombs, m. p. 59° ; 2 : 4-*dibromopropionanilide*, $C_6H_3Br_2{\cdot}NH{\cdot}CO{\cdot}C_2H_5$, colourless needles, m. p. 136°; *propionyl* 2 : 4-*dibromophenyl nitrogen bromide*, $C_6H_3Br_2{\cdot}NBr{\cdot}CO{\cdot}C_2H_5$, yellow rhombs, m. p. 87°; *propionyl* 2 : 4 - *dibromophenyl nitrogen chloride*, $C_6H_3Br_2{\cdot}NCl{\cdot}CO{\cdot}C_2H_5$, colourless plates, m. p. 71°; 2 : 4 : 6-*tribromopropionanilide*, $C_6H_2Br_3{\cdot}NH{\cdot}CO{\cdot}C_2H_5$, colourless prisms, m. p. 203°; *propionyl* 2 : 4 : 6-*tribromophenyl nitrogen bromide*, $C_6H_2Br_3{\cdot}NBr{\cdot}CO{\cdot}C_2H_5$, yellow prisms, m. p. 82°; *propionyl* 2 : 4 : 6-*tribromophenyl nitrogen chloride*, $C_6H_2Br_3{\cdot}NCl{\cdot}CO{\cdot}C_2H_5$, colourless prisms, m. p. 75°.

THE ROYAL SOCIETY.

Conversazione at Burlington House.

On Wednesday evening last (May 14, 1902) a Conversazione was held at the Society's Rooms, at Burlington House, and was largely attended by the Fellows and their guests.

The visitors were received by the President, Sir William Huggins, K.C.B., D.C.L., and the members of the Council.

Among the numerous and interesting exhibits, we may mention an improved form of Thomson Coal Calorimeter, exhibited by Mr. W. Rosenhain, in which the combustion chamber is specially designed to give easy manipulation, complete combustion, and complete transference of the heat of combustion to the water of the calorimeter. A special system of valves secures the thorough cooling of all escaping gases, by washing out the interior of the combustion chamber with the water of the calorimeter at the end of the combustion.

Mr. J. Mackenzie-Davidson exhibited some Stereoscopic X-Ray Transparencies and Negatives, two X-Ray Photographs of a bullet fired from a revolver, and, during the evening, showed the Development of Photographic Negatives in Ordinary Light, by which a reversed negative is obtained.

The Badische Anilin und Soda-Fabrik had an exhibit of Synthetic Indigo; they comprised—

(*a*) Specimens of the raw material (naphthalene) and the intermediate products formed in the manufacture of synthetic indigo, as well as of the latter in four different forms.

(*b*) Examples of various textile materials illustrating the application of synthetic indigo on loose wool, slubbing, military cloths of different nations, cops, cross-reeled bundles, cotton piece goods, both dyed and printed.

The initial material is naphthalene; this is converted by oxidation with sulphuric acid into phthalic anhydride, and the latter is next transformed into anthranilic acid, which, in conjunction with chloracetic acid, yields phenylglycin-ortho-carboxylic acid. The latter body is treated with a caustic alkali at a high temperature, and yields indoxyl or indoxylic acid.

It is interesting to note that this product indoxyl also results in the treatment of the indigo plant as practised in the tropics.

The last stage in the process, whether starting from naphthalene or from the indigo plant, is identical, viz., the oxidation of indoxyl into indigo.

An Apparatus for Natural Colour Photography, and examples of its applications, were shown by Messrs. Sanger, Shepherd, and Co. This process of natural colour photography is based upon Young's theory of trichromatic vision. Negatives or colour records of the object are taken through colour filters admitting light of the three primary sensations — red, green, and blue-violet — in accordance with the power of the respective sensations to excite the eye. From the three negatives three coloured positives are printed upon a special film, which, when mounted in superposition, exactly reproduce the form and colour of the object photographed.

There were also—

A New Camera for securing the three negatives through one lens at one exposure, and

A Camera for Photomicrographic Work fitted with colour filters for Natural Colour Photography.

Prof. H. A. Miers exhibited some Attempts to reproduce Polarisation Effects by Three-colour Printing. These are collotype prints from photographs of the coloured interference figures produced by crystal sections in a polariscope. The most successful of the reproductions is that of the figure yielded by a crystal of orthoclase felspar. The differences in the rings of the three monochromatic prints are in this case very noticeable.

Some Experiments illustrating the Effect of Ultra-Violet Light on the Electric Discharge were shown by Prof. J. A. Fleming. The effect of ultra-violet light in promoting electric discharge was discovered by Hertz in 1887 and has been much investigated since. The experiments exhibited show the action of the ultra-violet light emitted from the edge of the coating of a Leyden jar upon the spark of an induction coil charging it. The jar is arranged between two spark gaps in parallel, one larger than the other and both connected to the terminals of an induction coil. The jar is placed nearest to the shorter gap. The spark then happens always at the shorter gap. If a plate of glass or mica, both of which are very opaque to ultra-violet light, is then interposed between the jar and the active gap, a spark at once appears at the other gap. This spark can be quenched by illuminating the first or shorter gap with ultra-violet light from burning magnesium or a naked electric arc, and this can be done from considerable distances.

Dr. Dawson Turner also showed some Effects of Ultra-

Violet Radiation. Rock-salt appears to be the most transparent substance to this particular radiation.

Mr. W. R. PIDGEON exhibited a New Electrical Influence Machine suitable for Campaign Work. The sectors are imbedded in "volenite," and there is no glass or wax used in the machine, so that it will resist hard usage, even abuse, and when mounted on an iron frame is suitable for X-ray work in camp, or for work in India, and other countries, where the heat makes it difficult to preserve induction coils.

Prof. W. RAMSAY showed a Krypton Tube. Many persons see the colour of a vacuum tube containing krypton as lilac, many as green. The phenomenon appears to be conditioned by the size of the yellow spot on the retina.

A very simple Electricity Meter was shown by Messrs. G. L. FRICKER and W. MORDEY. It is suitable for both direct and alternating currents; is unaffected by changes of periodicity, and only requires to be wound once every three months or so.

Dr. A. MUIRHEAD gave a demonstration of Re-transmission on Submarine Telegraph Cables. In the cable relay the local contacts are made between a metallic arm, which is connected by fibres to the signal coil, and a metallic body whose surface is divided by their insulating partitions into three sections, to constitute the two terminals of a local line or re-transmitting battery, with a zero section between.

Two demonstrations, by means of the Electric Lantern, were given in the Meeting Hall: one at 9.45 p.m., by Sir H. TRUEMAN WOOD on the Application of Photography to the production of Pictures in Colour; and one at 10.45 p.m., by Dr. R. D. ROBERTS, who showed a number of Lantern Slides in Natural Colours, of the Grand Cañon of the Colorado, the Sierra Nevada, California, and the Yellowstone Park.

PHYSICAL SOCIETY.

Ordinary Meeting, May 9th, 1902.

Prof. S. P. THOMPSON, President, in the Chair.

DR. P. E. SHAW exhibited a Simple Electric Micrometer. Two years ago Dr. Shaw described an instrument with which he measured very small lengths by the application of electric contacts, and the micrometer shown was a simple form of the original apparatus. A screw, fitted with a milled head, turns in a fixed nut, and its lower end presses upon the extremity of the long arm of a lever. A metal point is attached to the short arm, and the distance through which it moves, on turning the milled head, can be deduced from a knowledge of the pitch of the screw, and the ratio between the arms of the lever. In using the instrument this point is always brought up to a metal surface, and the contact is accurately determined by the telephonic arrangement described in connection with the original micrometer. Dr. Shaw illustrated the use of the instrument for measuring small lengths by describing the following eight applications to ordinary laboratory measurements:—(1). The measurement of the thickness of plates, films, or fibres. The object is placed between two metal plates. The point of the micrometer is adjusted to touch the top plate, and the reading taken. The object is removed, the point is again brought into contact with the top plate, and the difference between the readings in the two cases gives the thickness of the film. (2). The determination of Young's modulus by the elongation of a wire. Dr. Shaw described experiments on two wires, each 2½ metres long, hanging side by side, one of copper and the other of steel. The wires terminated in horizontal platforms to which the stretching weights were attached. The base of the instrument rested on one platform, while depressions of the other, due to loading, were measured. In this way any error, on account of the bending of the beam from which the wires were hung, was eliminated. (3). The determination of Young's modulus by the bending of a beam. (4). The determination of simple rigidity by a static method. Observations were made upon a rod held horizontally by rigid wall brackets. One end of the rod was fixed, and the other held in position by a pin pressed into a hole in the end of the rod. From this end an arm projected outwards. Weights were applied to the extremity of this arm, and the twist measured by observing with the micrometer the movement of the end of the arm. (5). Application to the extensometer. (6). Measurement of thermal expansion. (7). Microscopic measurements. In measuring the diameter of a capillary tube the cross-wire of the microscope is made to touch one side of the tube, and the point of the micrometer is brought into contact with the metal stage. The stage is then moved by a screw until the cross-wire comes to the other side of the tube. The micrometer point is moved into contact again, and the difference in the readings gives the diameter of the tube. In this measurement the full magnifying power of the microscope is utilised, and the work of moving the stage is performed, by a rough screw. (8). The direct measurement of the wave-length of light. Newton's rings are formed by a convex lens and a piece of plate-glass. The convex lens is fixed to the short arm of the lever, and the distance through which it must be moved to cause a certain number of bands to appear at the centre gives a means of calculating the wave-length of the light employed.

Prof. EVERETT said the apparatus combined fineness and accuracy of measurement, and expressed his interest in the determination of distances with the microscope.

Mr. R. J. SOWTER asked if any special precautions had been taken to prevent deformation in the instrument. If not, then it was optimistic to assume that one or more multiplying levers could be used to measure lengths of the order of a millionth of a m.m. with any physical accuracy, assuming the multiplication of measurement to be according to the law of the lever as Dr. Shaw apparently did.

Mr. W. WATSON said that in determining the wave-length of light by Newton's rings it was necessary to measure from the knife-edge of the lever to the centre of the rings. The accuracy of the experiment was limited by the difficulty in judging the centre. The variation of the temperature of the air would distort the lever, and produce errors. In reference to the experiments on Young's modulus, using two wires of different material, he said that no precautions had been taken to procure a steady temperature, and pointed out that variations in temperature would affect the two wires unequally. In the experiment on the torsion of a rod the twisting had been produced by an unbalanced force. The other arm of the necessary couple must therefore have been unsatisfactorily supplied by the friction of the pin which held the rod in position. In measuring the diameter of a capillary tube with extreme accuracy it was necessary to have a normal section, which was somewhat difficult to obtain. Dr. Shaw apparently considered the instrument suitable for laboratory work. If it was for students it was unnecessarily elaborate, and if for obtaining accurate measurements there were objections and uncertainties which should be investigated. Mr. Watson said that a similar form of micrometer had been described by Mr. H. C. Russell, and used in the observatory at Sydney.

Mr. J. M. BARR said that as errors would be introduced by variations in temperature, it might be advisable to make the instrument of a nickel steel alloy with a small coefficient of expansion. He asked if the knife-edges and screws used were good enough to give the same reading twice, and said he thought the limit of physical accuracy could be obtained by using a single disc on a screw calibrated throughout its length. He considered one ten-thousandth of an inch to be about the limit attainable by such methods.

Dr. SHAW, in reply, said the main justification of the

apparatus was in the consistency of the results given by it; these were excellent, as the tables given in the paper showed. The method had been shown to be much more accurate than those in general use. As to the contention that the apparatus, while too sensitive for laboratory practice, was not sufficiently so for research on length changes, he pointed out that the accuracy of all measuring instruments was always on the increase, so that laboratory methods which were considered good recently would not necessarily suffice in the immediate future. Why be content, even for teaching students, with measuring to the one-fiftieth of a millimetre by the microscope when with the simple electric micrometer we get, with little trouble, to the thousandth of a millimetre? Dr. Shaw knew nothing of the prior publication in Australia mentioned by Mr. Watson; if there were such a method introduced years ago it was all the more astounding that Mr. Russell and his successors should have allowed such an excellent idea to lapse and remain immature and unknown. Some speakers had suggested that uncertain deformation of the levers might occur during an experiment and vitiate results; but a new strain implied a new stress, and unless a definite change occurred in the forces of the system we could not suppose a change in deformation. A like answer was given to the criticism that the levers might expand under heat during a measurement. Why if sufficient time has elapsed to allow the system to attain temperature equilibrium should anyone postulate expansions without indicating a new or irregular source of heat? Most of the above supposed evils of course existed, but they were of small account. As with every good apparatus ever produced, if ordinary and suitable precautions be taken, these errors were reduced to a lower order of importance, and could be neglected. In conclusion, the method had been firmly established in University College, Nottingham, and had superseded other apparatus. It would no doubt spread and become general.

Papers on "*The Conservation of Entropy*," by Mr. J. A. Erskine, and "*Rational Units of Electromagnetism*," by Signor G. Giorgi, were postponed; and the Society adjourned until May 23, 1902.

NOTICES OF BOOKS.

The New Volumes of the Encyclopædia Britannica. Constituting the Tenth Edition of that Work. The First of the New Volumes being Vol. XXV. of the Complete Work. London: *The Times*. Edinburgh: A. and C. Black. 1902. Pp. 808.

Whilst of considerable general interest a work such as this does not alone appeal to chemists and physicists, but equally to all groups of scientific men. This volume only extends to the end of the letter "A," and thus cannot comprise any great number of chemical subjects: we find a short article on Alloys, by Sir Wm. Roberts-Austen; one on Acetylene, by Vivian B. Lewes; Adulteration, by Otto Hehner; Aluminium, by E. Ristori; Aether, by J. Larmor; Argon, by Lord Rayleigh; and Assaying, by A. Blair. Each of these subjects is treated in the efficient manner we should expect considering the reputations of the experts who have undertaken the articles, and if the other branches of knowledge are dealt with in the same manner (and there is every evidence that this will be the case) we feel sure that these new volumes will ample fulfil the expectations of their publishers.

The Gas Engineer's Laboratory Handbook. By John Hornby, F.I.C. Second Edition, Revised and Enlarged. London: E. and F. N. Spon, Ltd. New York: Spon and Chamberlain. 1902. Pp. 312.

In revising this book it would have been as well if the author had entirely omitted the first thirty-nine pages which form Part I. The information given in this part is so thoroughly elementary that we can hardly conceive any gas engineer troubling to do more than glance at the pages and pass on. The very idea of explaining to the chemist at a gas works the difference between "qualitative" and "quantitative" analysis is absurd; the same may be said of the information given on pages 24 and 25, where the chemist is shown how to fold a filter-paper, and how to pour a solution out of a beaker into a funnel with the help of a glass rod; these instructions, by-the-bye, include three illustrations. Fig. 14 illustrates a "wash-bottle," and a very bad one at that; while on page 34 we find a picture of a pipe-clay triangle. What could be more unnecessary than to include such matter, which the veriest tyro should have at his finger ends?

The really useful portion of this book is in Part IV., on special analysis required in gas-works, and Part V., on technical gas analysis, calorimetry, estimation of cyanogen in gas, and the valuation of oils for gas-making purposes. Additional chapters have been added to the section on gas analysis; these include the description and use of the Orsat-Muencke apparatus, the method of determining the calorific power of gas, &c. This latter is of importance from the fact that heating by gas is coming more and more into general use.

The Orsat-Muencke apparatus is extremely useful for performing the technical gas analysis required in gas-works; it is applicable for the estimation of the absorbable constituents of such gaseous mixtures as crude coal-gas, water-gas, furnace and chimney-gases, &c. The apparatus is fully described and illustrated on pages 240—244.

In the appendix, which follows Part V., the Gas Referees' Instructions are given as to the times and mode of testing for purity, together with the useful and necessary tables for simplifying the calculation of results. The book is well indexed, and has a full table of contents.

Subject List of Works on Certain Chemical Industries in the Library of the Patent Office. London: Darling and Son, Ltd., and The Patent Office. 1901. Pp. 100.

This list, which is of undoubted value, is similar in form to the one already noticed (Chemical News, vol. lxxxv., p. 23), and unites under one cover the literature of certain organic industries usually associated in chemical literature. These subjects are divided naturally into five groups: —(1) Destructive Distillation Industries, including systems of lighting with hydrocarbons; (2) Industries of the Oils and Fats, viz., soap, perfumery, lubricants, candles, paints, and varnishes; (3) Industries of the Gums and Resins, including indiarubber and waterproofing; (4) Paper-making; and (5) Leather Manufacture. The present list comprises 1003 works representing some 1990 volumes, which are distributed under 151 headings and sub-headings.

The Electric Arc. By Hertha Ayrton, M.I.E.E. London: *The Electrician* Printing and Publishing Co., Ltd. Pp. 479.

In experimenting on the arc, the author's aim has been not so much to add to the large number of isolated facts already known, as to form some idea of the bearing these facts have one on the other. The attempt to correlate all the known phenomena, and to bind them together into one consistent whole, led to the deduction of new ideas which, when tested, became parts of the growing body of facts; in this manner the subject has grown and developed until it has reached its present magnitude and importance.

The theory which is presented in Chapter XII. has not been hastily built up to fit a few of the more salient characteristics of the arc, but has literally evolved itself during the course of a detailed study of each separate phenomenon. The central idea of this theory, that the carbon vapour changed into mist at a short distance from the crater dates from an early period of the author's work,

while the full recognition of all it entailed has followed slowly as each part of the subject was considered in turn.

The book is divided into twelve chapters, and contains 146 illustrations and 58 tables. The early chapters deal with the appearance and history of the arc, and the technical part of the work begins in Chapter III., on "striking" the arc, and sudden variations of current.

From Chapter IV. to Chapter IX. we have to do with current, electromotive force, potential difference between the carbons, &c., in all their varying bearings on the arc, its formation, behaviour, &c.

Chapter X. is on "hissing arcs," a subject which the author has made particularly her own, though much excellent work on this subject has been done by Trotter, Duddell, and others.

Chapter XI., which is an important one, is devoted to the consideration of the light and luminous efficiency of the arc; while Chapter XII., and last, on the mechanism of the arc, consists principally of matter embodied in a paper read before the Royal Society, June 20, 1901, on this subject.

This volume is a standing record of long and patient investigations, not of hurriedly put forward ideas, and shows careful weighing of all the evidence. At the end of each chapter there is a summary of the matter that has just been considered, wherein the author's conclusions are plainly and concisely set forth. The book is well and clearly printed, and the table of contents and index are satisfactory, while the short list of errata does not contain any correction of great moment; there is, however, one oversight to which we may draw attention: Chapter I. commences with the words, "Since the discovery of the electric arc early in the present century"; obviously a slip, which does not at all affect the high character of the book.

CORRESPONDENCE.

ARSENITE OF SILVER.

To the Editor of the Chemical News.

SIR,—I have read the curious letter signed A. G. Bloxam, which only becomes intelligible on the assumption that the writer of it was unacquainted with the contents of the admirable paper published by Bloxam in the *Journal of the Chemical Society* in the year 1862. One of the most interesting results published in that paper is the investigation of the zinc-salt of arsenious acid, the nature of which was made out in the most satisfactory manner. There are great difficulties in investigating the salts of such an acid as arsenious acid, and it reflects no discredit on Bloxam that he was not so fortunate with the silver-salt as with the zinc-salt.—I am, &c.,

J. ALFRED WANKLYN.

The Laboratory, New Malden, Surrey,
May 11, 1902.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxiv., No. 16, April 21, 1902.

Certain Phenomena of Voltaic Polarisation.—M. Berthelot.—Whilst examining voltaic action resulting from the reciprocal action of two liquids, phenomena of polarisation are often observed. These the author investigates, and especially the action of formic acid in such a case.

Absorption of Radio-activity by Liquids.—T. Tommasina.—Continuing his researches on the nature of the electric modification produced by radio-activity and the rapidity of appearance and disappearance of the phenomena in air, which happens almost immediately at the time of its maximum intensity, the author establishes the fact that the conductivity of liquid and solid dielectrics seems to increase under the action of radio-activity. The study of the absorption of the radio-activity is examined quantitatively, the substances experimented with being—carbon disulphide, toluol, benzene, essence of terebenthine, alcohol, oil of vaseline, petrol, distilled water, Rhone water, ammonia, concentrated solution of sodium bicarbonate, 10 per cent solution of sulphuric acid, 2 per cent solution of copper sulphate, acetic acid, lead acetate, amyl acetate, glycerin.

Formation of Negative Images under the Action of certain Vapours.—P. Vignon.—M. Colson's experiments in 1896 showed that zinc at ordinary temperatures emits vapours capable of altering photographic plates in the dark. Russell's experiments tend to the same result. The author now succeeds in obtaining images of medals dusted over with powdered zinc. These images are the negatives, not formed by the intervention of light and shadows, since they operate in total darkness. He also succeeds in obtaining negative images by allowing ammoniacal vapours to act on linen impregnated with a mixture obtained by grinding together powdered aloes and olive oil.

A Case of Molecular Rupture by means of Bromine.—R. Fosse.—The author hoped to obtain by the action of bromine on naphthyloldinaphthoxanthene,—

$$OH-C_{10}H_6-CH<\begin{matrix}C_{10}H_6\\C_{10}H_6\end{matrix}>O,$$

hydrobromic ether of naphtholdinaphthoxanthydrol formed according to the equation—

$$OH-C_{10}H_6-CH<\begin{matrix}C_{10}H_6\\C_{10}H_6\end{matrix}>O+Br_2=$$
$$=OH-C_{10}H_6-\underset{\displaystyle Br}{\underset{|}{C}}<\begin{matrix}C_{10}H_6\\C_{10}H_6\end{matrix}>O+HBr.$$

He finds, however, that HBr is not produced, but the bromine is fixed on to the molecule as if it were not saturated:—

$$OH-C_{10}H_6-CH<\begin{matrix}C_{10}H_6\\C_{10}H_6\end{matrix}>O+Br_2=$$
$$=OH-C_{10}H_6-Br+CH<\begin{matrix}C_{10}H_6\\C_{10}H_6\end{matrix}>O-Br.$$

Certain Derivatives of Fumaric Dialdehyde.—R. Marquis.—Acetine of nitrosuccinic aldehyde, described in a preceding paper, is decomposed by hot water at 80° into acetic acid, nitrous acid, and another product of aldehydic nature which remains dissolved in the water. Several derivatives of this product are prepared, its oxime and the benzoyl derivative of this, which his researches lead him to believe is a dialdehyde, and of constitution—

$$\begin{matrix}CH-CHO\\\|\\CH-CHO\end{matrix}.$$

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xxv., Nos. 20—21.

Sulphuric and Disulphuric Anhydrides.—G. Oddo.—Already inserted in full.

Acidimetry of Phosphoric Acid by means of the Alkaline Earths.—J. Cavalier.—Already noticed.

Oxidation of Propylglycol by means of the Oxidising Ferments.—André Kling.—Already noticed.

Action of Reducing Agents on the Two Isomerous Nitrodimethylacrylates of Ethyl.—L. Bouveault and A. Wahl.—Already noticed.

Nitroacetate of Ethyl.—A. Wahl.—Already noticed.

Action of Alcoholic Ammonia on the ω-Bromised Derivatives of Methyl-*p*-chlorophenylketone and Methyl-*p*-bromophenylketone.—A. Collet.—Twenty grms. of bromomethyl-*p*-chlorophenylketone were added to an excess (about 200 c.c.) of alcohol at 93° saturated with ammonia gas at the ordinary temperature; after being well shaken this is left to stand. The temperature rises slightly, and the liquid soon becomes of a golden-yellow colour. On the following day it deposits a reddish crystalline mass, which is collected on a filter and washed with cold water to eliminate the bromide of ammonium it may contain. In this manner about 6 or 7 grms. of a substance are obtained, which is purified by crystallisation in boiling acetic acid, forming yellowish crystals, which are transformed into almost colourless plates by a fresh crystallisation in chloroform. This substance has the composition of a di-*p*-chlorophenyl-γ-diazine, and its formula is $C_4H_2N_2(C_6H_4Cl)_2$; it is fusible at 200—201°, slightly soluble in boiling alcohol, more so in acetic acid, but especially so in chloroform. Bromomethyl-*p*-bromophenylketone, treated with alcoholic ammonia under the same conditions, gives a strongly coloured, deep reddish-brown liquid, which, after twenty-four hours, deposits a bright red crystalline mass (about 9 or 10 grms.). This deposit is purified by crystallisation in chloroform. It is di-*p*-bromophenyl-γ-diazine, $C_4H_2N_2(C_6H_4Br)_2$, fusible at 235—236°, slightly soluble in boiling alcohol and acetic acid and more so in chloroform. Both these diazines are soluble in boiling nitric acid (sp. gr. 1·40) and are deposited unchanged on cooling.

Pipette for the Determination of the Density of Liquids.—F. Girardet.—This paper requires the accompanying diagram.

MISCELLANEOUS.

The Royal Society.—The following are the fifteen selected candidates for election:—H. Brereton Baker, Prof. Henry T. Bovey, Prof. Rupert Boyce, John Brown, William Bate Hardy, Alfred Harker, Sydney S. Hough, Robert Kidston, Thomas Mather, John Henry Michell, Hugh Frank Newall, Prof. W. M. Flinders Petrie, William Jackson Pope, Edward Saunders, Dr. Arthur Willey.

Royal Institution.—On Tuesday next, May 20th, at three o'clock, Professor Karl Pearson will deliver the first of three lectures at the Royal Institution on "The Laws of Heredity, with special reference to Man"; and on Thursday, May 22nd, Mr. M. H. Spielmann commences a course of three lectures on "Contemporary British Sculpture," with lantern slide illustrations; and on Saturday, May 24th, Professor Brander Matthews begins a course of three lectures on "The Development of the British Drama." The Friday Evening Discourse on May 23rd will be given by Canon Ainger, his subject being "The Ethical Element in Shakespeare"; the Discourse on May 30th will be delivered by Mr. G. Marconi on "Electric Space Telegraphy;" and that on June 6th by Sir Benjamin Baker on the "Nile Reservoirs and Dams."

School Board for London.—Annual Exhibition of Handwork.—The Annual Exhibition of work executed in the Board Schools of London will be held at the Examination Hall, Victoria Embankment, W.C. (adjoining Waterloo Bridge), on Wednesday, June 18th, 1902, and the three following days. The Exhibition will be opened by Lord Reay, G.C.S.I., G.C.I.E. (Chairman of the Board), at 3 o'clock p.m., and will include specimens of Drawings, Colourwork, Modelling, Woodwork, Woodcarving, Metalwork, Needlework, Infants' work, Cookery, Laundrywork, Housewifery from the Day and Evening Schools, work from the Schools for the Blind, Deaf and Special Instruction, and also work from the Truant and Industrial Schools. There will also be included Scientific Apparatus which has been constructed by the teachers and pupils. The Exhibition this year should prove of particular interest, and it is hoped, inasmuch as admission to it will be entirely free, that the people of London will take this opportunity of seeing the really marvellous results obtained.

Oxylith.—The recent invention of "oxylith," due to M. Jaubert, will help to make the use of nascent oxygen still more wide in its applications. It consists of agglomerated peroxide of sodium which contains 15 litres of chemically pure oxygen (99·9 per cent) in every 100 grms. This oxygen is given off on placing the "oxylith" in contact with water, according to the equation—

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MEETINGS FOR THE WEEK.

TUESDAY, 20th.—Royal Institution, 3. "The Laws of Heredity, with special reference to Man," by Prof. Carl Pearson, M.A., F.R.S.

WEDNESDAY, 21st.—Microscopical, 7.30. Exhibition of Freshwater Entomostraca, by D. J. Scourfield.

THURSDAY, 22nd.—Royal Institution, 3. "Contemporary British Sculpture," by M. H. Spielmann.

FRIDAY, 23rd.—Royal Institution, 9. "The Ethical Element in Shakespeare," by The Rev. Canon Ainger, M.A., LL.D.

——— Physical, 5.

SATURDAY, 24th.—Royal Institution, 3. "The Development of the English Drama—I., The Art of the Dramatist," by Prof. Brander Matthews, Litt.D., D.C.L.

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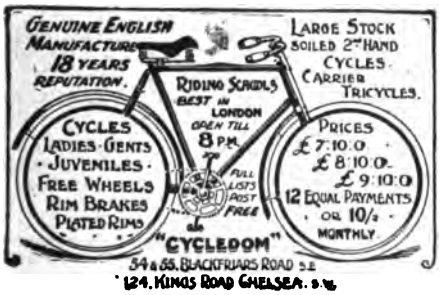
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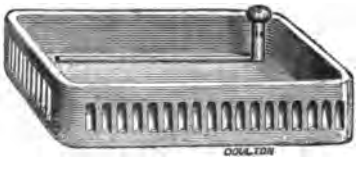

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AND

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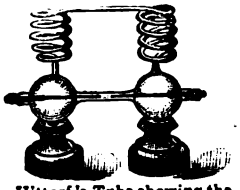

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THE CONDITIONS DETERMINATIVE OF CHEMICAL CHANGE AND OF ELECTRICAL CONDUCTION IN GASES, AND ON THE PHENOMENA OF LUMINOSITY.*

By HENRY E. ARMSTRONG, V.P.R.S.

In his communication to the Chemical Society on the union of oxygen and hydrogen, read at the meeting on February 19 (*Chem. Soc. Proc.*, 1902, 40), Mr. H. Brereton Baker has added another to his brilliant series of proofs that interactions supposed to take place between two substances are in reality of a more complex character; and having successfully demonstrated, in the case of the gases referred to, that water alone does not determine the interaction, he has, I believe, carried the investigation to the final stage which it was essential it should reach to make it a complete discovery of the nature of the process.

Mr. Baker has shown that when a mixture of hydrogen and oxygen [most carefully prepared by electrolysing a solution of barium hydrate] is enclosed in a chamber of hard Jena glass [cleansed in the most careful manner possible], and the mixture is dried as thoroughly as may be [by means of phosphoric anhydride which has been carefully purified by distilling it in a current of air], no appreciable interaction takes place between the gases, even on heating the tube to redness. If the drying be not carried too far, however, water is gradually but very slowly formed—finally in sufficient quantity to be visible; and yet, even when water is visibly present, no explosion takes place.

The interpretation I would give of these observations is as follows:—The amount of *impurity* present in the gases being reduced to a minimum, *i.e.*, the gases being *almost* dry and *almost* free from impurities *which in admixture with water constitute a conducting system*, change takes place at a *very* slow rate when heat is applied; and even when a considerable amount of *water* is present, the amount of associated impurity is too small to raise the conductivity—the rate of formation of conducting systems—to a point at which the rate of change would be such as to give rise to an explosive wave.

As defining the conditions which it has long been my opinion are necessary to the occurrence of chemical change in gases and generally, I may refer to words used by me in 1893.

("Eight years ago, in the course of the discussion on Mr. H. B. Baker's communication on "Combustion in Dried Gases" (*Roy. Soc. Proc.*, 1885, 40), I defined chemical action as *reversed electrolysis*; in other words, in order that chemical action may take place, it is essential that the system operated on comprise an electrolyte. I then pointed out that as neither hydrogen nor oxygen was an electrolyte, a mixture of only these two gases should not be explosive; and, moreover, that as water was not an electrolyte, and it was scarcely probable that water and [either] oxygen or hydrogen would form an electrolyte, it was difficult to understand how the presence of water pure and simple should be of influence in the case of a mixture of hydrogen and oxygen. This forecast has since been verified, the remarkable series of experiments carried out by V. Meyer in conjunction with Krause and Askenasy having clearly demonstrated that the formation of water from hydrogen and oxygen takes place at an irregular rate, and is, therefore, dependent on the presence of a something other than water—I imagine an acid impurity. But this is a consideration which has not yet received the proper attention, and it is, therefore, desirable to emphasise its importance by reference to other cases. Mr. Baker's recent preliminary note on the influence of moisture in promoting chemical action (*ante*, p. 229) affords several interesting examples:—Thus, he states that neither does hydrogen chloride combine with ammonia nor is nitric oxide oxidised by oxygen if moisture be excluded. In the former case, the addition of water should suffice to determination the combination, as water and hydrogen chloride together form a 'composite electrolyte' (*cf. Roy. Soc. Proc.*, 1886, No. 243, p. 268); as neither nitric oxide nor oxygen, however, forms a composite electrolyte with water, in this case water alone should not determine the occurrence of change—but if by the introduction of a trace of 'impurity' in addition to water the presence of a composite electrolyte were secured (however high its resistance, owing to the smallness of the amount of 'impurity'), action would set in, and when once commenced would proceed at an increasing rate, as nitric acid would be formed, and the resistance of the electrolyte would consequently diminish. On this account it will be a task of exceeding difficulty to demonstrate experimentally that nitric oxide and oxygen are inactive in presence of water alone; but there can be no doubt that such must eventually be admitted to be the case, provided always that it is permissible to extrapolate Kohlrausch's observations, and to conclude from them that *pure* water is a dielectric."—*Chem. Soc. Proc.*, 1893, 145).

I venture to call attention to them, not because I have any particular wish to put forward a claim on my own behalf, but in the hope of attracting attention to a subject of surpassing importance, which both chemists and physicists have hitherto most strangely neglected to consider *in all its bearings*.

[In his complete paper (*Chem. Soc. Trans.*, 1902, p. 400), Mr. Baker states that he found that hydrogen and oxygen did not interact when subjected to the influence of a coil of thin silver wire, heated by an electric current to the melting-point of the silver, that is to say, above 1000°; but that when a similar platinum coil was tested in the dried gaseous mixture, an explosion occurred just after the wire became visibly red. Doubtless, in the latter case, the surface over which interaction extended was sufficiently large to raise the velocity of change to the explosive rate, owing to the condensing effect exercised by the platinum; and the silver had no effect, because it is destitute of the power which platinum possesses in so high a degree of attracting gases and of acting as a catalyst.

In making this statement, I wish it to be understood that I assume that platinum *per se* would be without effect. I feel almost confident, in fact, that if they could be dealt with, even free atoms would not associate in the absence of a composite electrolyte, as it appears to me probable that at least one function of the composite electrolyte is to provide the "mechanism" by means of which the energy of chemical change is frittered away. Ostwald, in his lecture on "Catalysis" (an English translation of this is to be found in *Nature*, April 3, 1902), defines a catalyst as any substance which alters the velocity of a chemical action without appearing in the final product. I conceive, however, that the catalyst determines the interchange from its beginning. I have discussed a number of "catalytic" phenomena—including those of fermentation and of the dissolution of metals in nitric acid—in the address referred to later on.—*Noté, added May 2*].

It appears to me that Brereton Baker's results lead to far-reaching consequences—that they justify, not only the conclusions I have drawn as to the conditions which determine the occurrence of chemical change, but also the conclusion already stated by me in the note previously referred to, in 1893, that *pure* gases should be perfect dielectrics; *i.e.*, that the passage of an electric discharge through a gas, like that of an explosive wave through, say,

* A Paper read before the Royal Society, May 1, 1902.

a mixture of hydrogen and oxygen gases, can only take place if an electrolyte be present, such electrolyte being, it would seem, always a composite system, and one which may be pictured as existing momentarily in a quasi-liquid state.

The argument was more fully developed in my Presidential Address to the Chemical Society, in 1895, in which I discussed very fully the nature of chemical change, and the conditions which determine it, from various points of view.

(No more striking evidence that the occurrence of a discharge in a gas is dependent on the presence of a something besides the gas—on the formation of a complex conducting system—could be given than is afforded by the beautiful experiment exhibited by Dewar at the Royal Institution, showing that the discharge at once ceases in one of Crookes's phosphorescent tubes on cooling with liquid oxygen. Such treatment cannot be supposed to cause the condensation of the residual air, but may well condition the deposition of the traces of vapour (? of conducting water) present in the gas, thereby destroying the systems within which conduction can alone occur. It may be here pointed out that this argument is perhaps also at least partially applicable in explanation of the erratic behaviour of the discharge in vacuum tubes. If the discharge occur within a complex system, it may well be that certain conditions will favour the formation of a conducting system including one, and others of a system including another, of several substances present in a tube—such an explanation, for example, would account for the fact that the mercury spectrum is only sometimes seen in tubes connected with a mercury pump. And having regard to the fact that a gas may exhibit several spectra, it may be that the spectrum of a given substance varies more or less according as it is included in one or other of several conducting systems.—*Chem. Soc. Trans.*, 1895, 1141).

No attempt has been made as yet to deal practically with the problem of electrical discharge in gases in the way in which Dixon, Brereton Baker, and Shenstone have dealt with that of chemical change. Vacuum tubes are invariably made of soft glass—which is altogether unfitted for accurate experiments, as it is always more or less readily attacked by moisture, yielding up traces of alkali; no special care is taken in cleansing them; no special precautions are used in filling them; and both oil of vitriol and *commercial* phosphoric anhydride are used as drying agents, although it is well known that these may be fruitful sources of contamination. And the electrodes offer special difficulties, which it may be impossible to overcome, owing to the readiness with which metals occlude gases.

If there be evidence to show that the discharge in vacuum tubes is conditioned by the presence of impurities, as I contend there is, it is open to question whether most, if not all, of the effects attributed to the so-called radiant matter—or as Sir William Crookes now expresses it, to electrons—are not in reality due to ordinary gross matter. To take an example from this author's recent communication to the Society on "Radio-activity and the Electron Theory," at the close of the paper reference is made to the discharge from a silver pole of *electrons* which cause the glass against which they strike to glow; at the same time, the silver volatilises and is deposited near the pole. "While the volatilisation of the pole is rapidly proceeding, the metal glows as if red hot. This 'red heat' is superficial only." It appears to me possible to give a more ordinary explanation of the remarkable phenomenon thus described by Sir William Crookes. A silver electrode would be more or less "polarised" with oxygen—for we know that silver has the power of absorbing oxygen when fused, and that the gas is only partially extruded as the metal cools; in any case a minute amount of oxygen would be present in the tube. When heated by the discharge *in vacuo*, the silver-oxygen "compound" would dissociate—*i.e.*, oxygen would be given off, but would be in part re-absorbed, so that the silver would never be entirely free from oxygen; the change, in fact, would oscillate about a point of equilibrium, and would never be total in the direction corresponding to the complete deoxidation of the silver. May not the bombardment of the glass described by Sir William Crookes have been effected by the displaced oxygen molecules, and may not the surface heating of the pole have been an outward and visible sign of the return of the truants to their silvery home?

A diamond pole, such as Sir William Crookes speaks of, might behave in a similar manner in presence of a minute amount of oxygen; in fact, the argument applies generally to the occlusion of hydrogen and other gases by electrodes.

An argument which I think will sooner or later be regarded as of weight in favour of the view that the phenomena are electrolytic in their origin, is afforded by the luminous manifestations in vacuum tubes. These can scarcely be either mere collision effects, or mere heat effects. It has long seemed to me that luminosity and line spectra are the expression—the visible signs—of the changes attending the formation of molecules from their atoms, or, speaking generally, *that they are consequences of chemical changes*, a chemical change being one which involves an alteration of molecular composition, or it may be of molecular configuration, as it is conceivable that even changes involving but the formation of isodynamic (tautomeric) molecules—changes in molecular structure unattended with change in molecular size—may give rise to such manifestations. Dealing with the question of the luminosity of hydrocarbon flames, I expressed this view in 1895 in the terms given below.

("Interest should be revived in the subject by the recent observations of V. B. Lewes (*Roy. Soc. Proc.*, 1895, vol. lvii., p. 450) on the decomposition of acetylene at high temperatures, which have led him to suggest that this compound plays the chief part in promoting luminosity in hydrocarbon flames, and that the heat liberated during its decomposition endows the carbon particles produced from it with an incandescence far higher than corresponds to the temperature of the flame. It appears to me that while accepting this as a partial explanation, it is unnecessary to suppose that luminosity is consequent on the production of presumably solid carbon particles; the conversion of acetylene, or other hydrocarbons, at high temperatures into hydrogen and carbon, although a decomposition in the ordinary sense of the term, is doubtless a change involving the interaction of several molecules, and the consequent formation of carbon molecules of a high order of complexity together with hydrogen molecules; and it is exothermic because the energy liberated in the combination of the carbon atoms among themselves, and also of the hydrogen atoms, is far in excess of that absorbed in the decomposition of the hydrocarbon molecules. The initial luminance may therefore well be that of the molecules at the moment of formation prior to condensation, although the continued incandescence of the carbon molecules after their reduction to the solid state probably contributes largely to maintain luminosity, giving to hydrocarbon flames their special value, perhaps. But this is to explain the phenomena in terms of Frankland's theory, as I understand it, and as appears to be necessary in order to explain the luminous appearance of a hydrogen-oxygen flame burning under pressure, and other cases in which the products of combustion are gaseous."—*Chem. Soc. Trans.*, 1895, p. 1147).

In the discussion on Mr. Campbell Swinton's paper on the luminosity of the rare earths when heated *in vacuo* by means of cathode rays, read in April, 1899 (*Roy. Soc. Proc.*, vol. lxv., p. 115), I applied this view in explanation of the brilliant luminosity of certain rare earths, and also of the electric arc, suggesting in the latter case that the carbon molecules were dissociated by the passage of the

current, and that the re-association of the atoms into molecules gave rise to the intensely luminous effect which is characteristic of the arc.

Bunte has argued (*Deut. Chem. Ges. Ber.*, 1898, p. 5), in the case of the Auer von Welsbach gas mantle, that the high luminosity is not so much due to a specific superior radiating power of the earth used as to the fact that, in virtue of the power which a substance such as ceria possesses of forming a peroxide, combustion takes place to a greater extent at a surface on which it is present than on one consisting of a neutral oxide; consequently, the temperature is higher at such a surface. This undoubtedly must be the case; but I would go further, and regard *the chemical changes* occurring at the surface as the direct seat, or origin as it were, of the luminosity. Probably, a higher oxide is alternately decomposed and reformed—in other words, *the process is one of oscillatory or re-current oxidation*. Owing to the influence which the oxide exercises as a catalyst combustion doubtless takes place at its surface more completely than it would at a corresponding neutral surface, and in consequence the temperature developed at the oxide surface is above that of the flame generally.

[It is noteworthy that besides ceria, which is by far the most efficient, only one or two other oxides are effective excitants; and that cerium dioxide is remarkably stable at high temperatures.—*Note, added May 2*].

A similar explanation may be applied to the incandescence of oxides generally, such as is witnessed, for example, in the lime and zirconia lights. The Nernst lamp probably owes its efficiency to a like cause. It may well be that in the case of the incandescent mantle the maximum effect is produced when only a relatively small proportion of active oxide is present, because at the particular state of dilution the oxide is present in solid solution in a form in which it is most prone to suffer change—that besides being placed under the necessary conditions of freedom, it attains to the most suitable degree of molecular complexity, and therefore to its greatest activity in dilute solid solution, just as many substances do in dilute liquid solutions.

Mr. Campbell Swinton's observations appear to me to be in harmony with these conclusions. An intensity of cathode rays that gave a brilliant light with "pure" thoria and with a mixture of 99 per cent thoria with 1 per cent ceria was found by him to give practically no light with "pure" ceria and with a mixture of equal parts of thoria and ceria. It is improbable that Mr. Swinton dealt with *pure* thoria, as the methods of purification practised at that time did not suffice to give a pure product. That thoria alone gave more light under the influence of cathode rays than in the Bunsen flame may have been due to the fact that it was more intensely heated in the former case, the effect produced by minute proportions of the exciting oxide being much dependent on temperature, and greater the higher the temperature.

The need of an explanation such as is here given of the luminosity of oxides at high temperatures has, I know occurred to others. My colleague, Professor Ayrton, has long held such a view, and he informs me that, in the course of conversation with him in 1897, Professor Elihu Thomson gave it as his opinion that the brilliant incandescence of lime in the oxy-hydrogen flame was not a mere high temperature effect (compare *Journal of the Society of Arts*, February 10, 1899, p. 256; E. Pringsheim, International Physical Congress at Paris, "Report 2").

The phenomena of phosphorescence under the influence of the electric discharge exhibited by the oxides and basic sulphates of the rare earths, with which Sir William Crookes's classical researches have made us familar, may be included in the same category with those above discussed; if not due to recurrent oxidation, they may be cases of recurrent polymerisation. Apparently the same oxides act as excitants whether a flame or cathode rays be used. Sir William Crookes, it is well known, holds the view that yttria, lanthana, &c., are characterised by definite phosphorescent spectra; on the other hand, Lecoq de Boisbaudran has contended that the yttria and gadolinite earths are not self-luminous. This latter view has recently been confirmed by Baur and Marc (*Deut. Chem. Ges. Ber.*, 1901, p. 2460). According to these observers, whilst the colourless oxides and salts of yttrium, gadolinium, and lanthanum are not specifically luminescent, if mixed with minute proportions of the earths which have coloured salts, viz., erbium, neo- or praseodymium, they afford spectra such as have hitherto been regarded as characteristic of themselves; and such spectra may be equally well developed by using lime or calcium sulphate as diluents; yttria itself, however, fails to give a spectrum when mixed in small proportion with lime.

[If it should eventually be established that the emission spectra are conditioned solely by "earths" which afford coloured salts, as the emission spectra appear to be closely related to the absorption spectra, dark lines in the one taking the place of bright lines in the other, it will be possible to correlate the production of visible colour with that of a luminous spectrum, and to regard both as originating in the same intramolecular mechanism.—*Note, added May 2*].

The argument may be carried a stage further and applied to phosphorescent phenomena generally. It appears possible to regard all these as the outcome of oscillatory changes in molecular structure—and the slow decay of the effect which is observed in many cases may be compared with the slow discharge of a condenser—the charging of which be it noted may be regarded as but a process of molecular deformation of the dielectric.

(To be continued).

HISTORY OF THE SYNTHESIS OF INDIGO.*

By AD. BAEYER.

HAVING been asked to give a history of the synthesis of indigo, I would first remark that I have already published, twenty years ago, an account of my work on indigo; at that time I wrote as follows (*Berichte*, vol. xiii., p. 2254):—"The discovery of an easily performed synthesis of indigo blue (starting with chlorised isatin) having closed an important chapter in the history of this colouring material, I take the opportunity of glancing over the course of my research which has led to this result. This research is described in a large number of isolated notes not having any apparent connection, and I think I can be of service to my colleagues by showing, on the one hand, that the discovery of artificial indigo has been the fruit of a long series of experimental researches, systematically and intimately connected together, and by particularising, on the other hand, the line of work I am adopting at the present time based on the facts discovered by myself and my pupils."

Indol, extracted for the first time from indigo, acquired great importance in physiological chemistry from the fact of its formation in the animal organism.

Isatin, discovered simultaneously by Erdmann and Laurent, furnished aniline to Hofmann by fusion with potash, and enabled him to prepare the mono- and dichlorised, and mono- and dibromised anilines. It was, further, the starting-point of my work on indigo. In the paper just quoted the following words are to be found:—"The first problem I set myself was to eliminate the oxygen from isatin, and we know how it was solved in 1865 and 1866 by the transformation of isatin into dioxindol, oxindol, and indol. The enormous expense of time and work entailed by the discovery of the method of reduction by zinc powder, a method necessary to realise

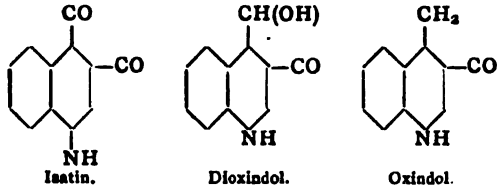
* Abridged from the Report of the Conference held on the occasion of the Inauguration of the Hofmann Institute at the German Chemical Society.

the desired end, was further recompensed by the discovery in 1868 of *artificial alizarin* in my own laboratory by my assistant Graebe, and M. Liebermann."

While studying one day the properties of isatin, I was struck by the great analogy which existed between it and alloxane. The reduction of isatin, made by Laurent and Erdmann in 1840, did not go beyond the first phase, isathyde, as the other derivatives discovered by these chemists, indene, hydrindene, &c., were only complex products of condensation.

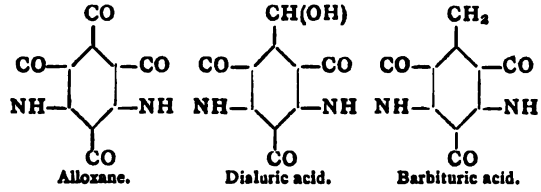
But there was a hope that the chain of thought which had led to malonyl urea would also be of service in the case of isatin. This hope was realised when, in 1865, I took up the research on isatin with my pupil C. A. Knop. By successively treating isatin with sodium amalgam and tin and hydrochloric acid, we obtained dioxindol corresponding to dialuric acid, and, finally, the ultimate term of the series corresponding to barbituric acid, viz., oxindol.

Isatin Series.



Isatin. Dioxindol. Oxindol.

Alloxans Series.



Alloxane. Dialuric acid. Barbituric acid.

The above formulæ for alloxane were adopted from my work on uric acid, and still hold good.

But an idea occurred to me during the research which prevented me from recognising that the derivatives of isatin were constituted in a very analogous manner. The phenolic properties of oxindol led me to consider it as the phenol of a hypothetical compound, to which I assigned the formula—

$$C_6H_4<\begin{matrix}CH_2\\N\end{matrix}>CH,$$

and to which I gave the name of *indol*. I immediately attempted the preparation of the hypothetical indol. I started with oxindol which only contained one atom too much oxygen, and which behaved like phenol. After six months of unsuccess I confided my trouble to my colleague Stahlschmidt, and from him learned that zinc powder had been for some time used as a reducing agent. I at once made the experiment, but the oxindol resisted all my efforts, until in despair I heated some almost to redness with zinc powder in a combustion tube. At last I had in my hands the substance from which indigo is made.

In my paper in 1880 I wrote:—"Henceforth all my efforts were turned to the discovery of a simple method for the preparation of indol, as I considered it as the true source of the indigotic group. In 1868 a direct method was found for obtaining indol by the action of zinc powder on indigo blue, and in 1869 a synthetical method starting from nitrocinnanic acid and its derivatives (*Berichte*, vol. ii., p. 679). This latter paper contains a passage which I quote in full as it contains the complete programme of my work on indigo up to the present time: 'If we wish to prepare indol synthetically it is necessary, according to the above formulæ, to introduce a lateral chain into the benzenic radical, formed of two carbon links, and one atom of nitrogen, and to join them together. The conditions necessary for effecting this operation are found in nitrocinnamic acid, if we abstract the carbonic acid and the oxygen from the nitrated group. And, in fact, we have found that when fused with potash, nitrocinnamic acid gives indol.'"

For convenience of handling, I have now arranged my materials, not in chronological order, but in order of importance, viz., indol, isatin, indigo. The study of indol helped to found the theory of the closure of chains, that of isatin to establish desmotropic isomerism, and the work on indigo led to a whole series of new methods which were utilised later on.

The History of Indol.

Before this substance was discovered I gave it the following formula:—

$$C_6H_4<\begin{matrix}CH_2\\N\end{matrix}>CH.$$

When I had obtained it, I noticed a great analogy between indol and pyrrol, and endeavoured to determine the constitution of this latter body (*Berichte*, vol. ii., p. 99). We discovered pyrrolate of potassium, and from it deduced the formulæ of pyrrol, indol, and furfurane. My synthesis of indol was, however, interpreted in a different manner by other chemists. Up to 1879, indol had only been prepared by using high temperatures, but during this year I found a method which enabled me to obtain indol by the wet way. After the transformation of isatin, by the action of pentachloride of phosphorus, into chlorised isatin, and the conversion of this latter, by reduction, into indigo, I submitted oxindol to the same treatment. The first reaction was easily obtained, but dichlorised indol was formed instead of the monochlorised product.

The substitution of hydrogen for the chlorine offered the greatest difficulties, and could not be effected by zinc powder, or hydriodic acid, on account of resinification, while sodium amalgam or sodium in etherial or alcoholic solution would not attack the chlorine. But by heating the dichlorised product to boiling in alcoholic or, better still, amylic solution, and introducing small pieces of sodium, I succeeded in attaining my end. This led to the synthesis of quinolein from cinnamic acid, and thus revealed the relation between indol and quinolein. About the same time the law governing the closure of chains was recognised which is at the basis of this synthesis.

The researches on oxindol, containing two atoms of oxygen in the lateral chain, led me to submit to the same treatment bodies of the same nature, but containing three atoms of carbon, carbostyrile and hydrocarbostyrile. The latter easily gave a dichlorised quinolein corresponding to the twice chlorised indol; but with a greater number of carbon atoms in the lateral chain the cyclic closing could not be obtained.

My attempts to prepare orthonitrophenylacetic aldehyde by the method indicated in a note published in 1880 were unsuccessful, but, on the other hand, they led to the discovery of the synthesis of indigo, starting from orthonitrocinnamic acid.

Contribution to the History of Isatin.

By the reduction of isatin to indol, I gained the certain knowledge that this class of body was formed by the products of the oxidation of indol. I considered isatin to be an orthoquinone, although at that time no compound of this class was known, and at the same time I gave the formulæ of a pyrocatechin, and a phenol derivative, to dioxindol and oxindol; this turned out to be quite correct.

Soon afterwards Kékulé criticised my theories, and submitted the hypothesis that indol was an orthoaminophenylacetylene, dioxindol an aldehyde, isatic acid an aminised derivative of benzolylformic acid, and isatin an anhydride of this acid. He gave the following formulæ to isatic acid and isatin:—

$$C_6H_4<\begin{matrix}CO.COOH\\NH_2\end{matrix}$$ Isatic acid.

$$C_6H_4<\begin{matrix}CO.CO\\NH\end{matrix}>.$$ Isatin.

His conception of isatic acid and the relations between it and isatin were quite new. It showed a way for the synthesis of isatin, on which he expressed himself as follows:—

"I am engaged in transforming phenylacetic acid, first into bromophenylacetic acid, and then into nitrobromophenylacetic acid. By the reduction of this latter there will be formed probably orthoamidophenylacetic acid, and at the same time a body corresponding to carbostyril. If we succeed in oxidising this acid in such a manner that the hydrogen of the lateral chain is replaced by oxygen, we shall obtain isatic acid and isatin."

Kékulé wished, by the introduction of bromine, to put the nitro group into the ortho-position, but his pupil was not able to effect this.

Eight years elapsed after Kékulé's first publication without any appreciable results being obtained, when I undertook to test the truth of his ideas. I said to myself that if isatin was really the anhydride of isatic acid, the products of the reduction of the latter would be to the products of the reduction of isatin as the acid itself is to its anhydride.

As isatic acid is only stable in alkaline solution and cannot be reduced under these conditions, we first acetylised it, and then reduced the stable acetylised derivative in acid solution by means of sodium amalgam in acetic solution. In this manner acetylhydrindic acid was obtained, and by the further reduction of this *oxindol*. The slightly enlarged conception of Kékulé having been thus confirmed, we were able to proceed to the realisation of his programme for the synthesis of isatin.

Profiting by a remark of Radziszewski, that the ortho-nitrated acid forms in large quantities at high temperatures, I conducted the nitration on the boiling water-bath, and obtained a good return of the substance so long sought for in vain. As could be foreseen, the reduction of this acid gave synthetic oxindol, and the amido-oxindol prepared from this latter was easily transformed by oxidation into isatin. Thus, on June 6, 1878, I realised, for the first time, the synthesis of isatin. The transformation, discovered in 1870, of isatin into indigo was thus completed, and finished the entire synthesis of the colouring material.

During the next year, my work produced a new product of the reduction of isatin, viz., hydroïsatin. The solution of isatin and of its acetylised derivative in glacial acetic acid is decolorised by zinc powder; water precipitates a white substance, which, when exposed to the air, is again transformed into isatin. But if we boil an aqueous solution of isatin, treated with a little hydrochloric acid, with zinc powder, after a short while there is a persistent decoloration, with the formation of oxindol. Hydroïsatin should therefore be considered the first product of the reduction of isatin.

The transformation of Kékulé's isatin into my hydroïsatin corresponded to the reduction of phenanthrene quinone into dioxyphenanthrene. With the di-ketonic formula it was difficult to conceive that the two atoms of oxygen could be reduced simultaneously with the formation of a double bond which had not previously existed. A special hypothesis was necessary according to which the two ketonic oxygens in the ortho- and para-positions should influence each other mutually. The merit of having raised this hypothesis to the position of a general law is due to J. Thiele.

I had observed, with Rupe, that in the reduction of muconic acid, the two atoms of hydrogen were fixed to the ends of the hydrocarbonised chain contained in this acid, and, at the same time, a new double bond was formed in the centre of the chain.

I explained this particular phenomenon by the attraction the carboxylised groups had for the atoms of hydrogen. Thiele put forward the hypothesis that two double bonds, close to each other, were possessed of a particular structure which required a new symbol to express it. A pair of double bonds could be compared to a magnet which is only active at its extremities; when cut in half it forms two magnets, and when made into a ring it loses its external power of acting. By admitting this as true, hydroïsatin should be looked upon as an isomer of dioxindol; that is to say, as the true dioxindol corresponding to pyrocatechin. The substance which had before received the name of dioxindol results from the transposition of hydroïsatin, according to Erlenmeyer's rule, and it is to this transposition, which it was impossible to foresee, that we must attribute the fact that the nature of this class of compounds remained obscure for so long.

Researches carried on with Oekonomides in 1882 showed that in the ethers of isatin, prepared by means of the silver salt, the alcoholic radicals were fixed to the oxygen, while acetyl-isatin could only contain the acid radical joined to the nitrogen. Consequently, isatin reacts in two different manners, a phenomenon called tautomery by Laar later on; these two forms I called *lactame* and *lactime*, and I consider that it is most probable that in the solid state isatin is a lactame, while it does not appear to me to be impossible that the solution may also contain a lactime modification.

In 1883 I published this theory with regard to indoxyl, and expressed myself as follows:—"Isomers are only known in the combined state; in the free state they return spontaneously to their initial form. Their instability should be attributed to the mobility of the hydrogen atoms, assuming that the substitution of other groups for the hydrogen renders the compounds stable."

Contribution to the History of the Artificial Preparation of Indigo.

Artificial indigo first saw light in the year 1870, when, in collaboration with Emmerling, I succeeded in transforming isatin into indigo blue by the action of trichloride of phosphorus containing free phosphorus. As, at this time, isatin could only be prepared from indigo itself, this synthesis did not become complete until I succeeded on June 6, 1878, in preparing isatin artificially from phenylacetic acid. Shortly after my first publication Emmerling and Engler observed traces of indigo formed from materials not originally made from the colouring matter, by heating the syrupy mass obtained as a by-product in the nitration of acetophenone, with zinc powder and soda lime, by the method I used for the transformation of nitrocinnamic acid into indol. They were of the opinion that indigo was an azoic colouring matter, and that seemed experimentally correct, but I looked upon indigo as an oxygenated derivative of indol. As they obtained a trace of indigo, although with very great difficulty, the way seemed open for its synthesis from acetophenone.

Later on, however, they were unable to repeat their experiment, and six years later they wrote:—"We frankly state that one of us has not succeeded—after repeating the nitration experiment innumerable times with the most diverse kinds of fuming nitric acid, and under the most varied conditions—in obtaining a syrupy nitrated product, which, like that previously prepared, would remain syrupy for several weeks; and it is exactly this syrupy product that we require as a starting point. . . . . In face of the refutation of our synthesis of indigo blue by Wichelhaus we are powerless, inasmuch as, in spite of reiterated experiments, we have not been able to succeed in reproducing the conditions under which we obtained the indigo, and that the method in question did not enable us to produce the indisputable proof of our synthesis; that is to say, to prepare a quantity of the substance for analysis."

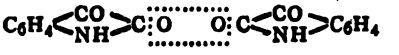
The merit of having discovered a synthesis of indigo is due, I think, to Nencki, who, in 1875, observed that indol, suspended in water, gave traces of indigo when treated with ozone.

The considerations which led me, thirty years ago, to the preparation of artificial indigo from isatin were the following:—"One of the most important problems of the chemistry of indigo is the preparation of this colouring matter from isatin. Although isatin could only be reduced

to the state of isathyd, there were hopes that the problem might be solved by the further reduction of isatin. But the work done in collaboration with Knop made known a whole series of products of reduction of isatin, and we even succeeded in removing the whole of the oxygen from isatin without obtaining a single reaction leading to indigo blue. The hope of effecting the synthesis of indigo seemed therefore to become less and less. If we consider the nature of the reagents employed up to the present for the reduction of isatin, we see that they are all *hydrogenating* agents, and that there has not been a body used capable of removing the oxygen from the isatin without hydrogenating this latter. This method alone offered some chance of success, and, in fact, experiment showed that for the reduction of isatin into indigo blue it was necessary to totally exclude hydrogen, as the action of this element led to a series of entirely different compounds which could not be transformed into indigo. A reagent which realises these conditions is trichloride of phosphorus containing free phosphorus."

These considerations were fully confirmed by experiments made later on. In the reduction of isatin to oxindol, it is precisely the atom of oxygen necessary for the formation of indigo that is replaced by the hydrogen. On the other hand, the phosphorus leaves this atom of oxygen intact, and, by eliminating the other atom of oxygen from the isatin, leads directly to the formation of indigo, as the following formulæ show:—

Isatin. Oxindol.



Indigo.

In the years 1878-1879 I found that the synthesis of indigo could be improved by starting with chloride of isatin; the reduction of this body probably depends on the addition of two atoms of hydrogen with the formation of chlorised indoxyl, which is transformed directly into indigo by the loss of hydrochloric acid:—

This indoxyl was discovered simultaneously by Baumann and Tiemann in 1879, and oxindol should be considered even more as the true mother-substance of indigo, as its formation always preceded that of indigo in nearly every synthesis.

Baumann first called attention to the fact that indicone from urine was different to the vegetable indicone obtained by Schunck. Later on, in collaboration with Brieger, he showed that indicone contained a substance named indoxylsulphuric acid, in the examination of which he was associated with Tiemann.

These two chemists then found that, as with phenolsulphate of potash, indolsulphate of potash was split up by acids into sulphuric acid and a body of a phenolic nature, indoxyl. Treated with an oxidising agent, indoxyl was transformed almost quantitatively into indigo. I found the same properties for indoxyl prepared later on synthetically. The formula they assigned to indoxyl is still used; they rightly compared the transformation of indoxyl into indigo with the transformation of dimethylpyrogallic ether into cerdriret, but they placed the double bond produced by the formation of the colouring material in the benzenic instead of the pyrolic radical, and thus arrived at a formula for indigo which does not agree with the properties of this body.

To them, however, is due the merit of having discovered indoxyl and showing its easy transformation into indol. I, on the other hand, did not agree with them at first, and only recognised the truth of their observations two years later, after having prepared indoxyl synthetically, and established its identity with that obtained from urine.

The idea of using the synthesis of indigo commercially did not arise till 1880, when I commenced using cinnamic acid instead of phenylacetic acid.

In the course of an experiment instituted with a view to prepare orthonitrophenylacetic aldehyde, I boiled the bromide of orthonitrocinnamic acid with alkalis, and found that a small quantity of indigo was formed. The study of this reaction led me to the discovery of orthonitrophenylpropiolic acid and to the transformation of that body into indigo. This invention was patented on March 19, 1880, and was published for the first time in December of the same year.

My researches were considerably helped by the numerous works on cinnamic acid published during the period between 1860 and 1880. The extensive researches of Glaser, Beilstein, and Kuhlberg on orthonitrocinnamic acid were particularly useful to me.—*Moniteur Scientifique*, Series 4, vol. xv.. p. 145.

CHEMICAL SOCIETIES OF THE NINETEENTH CENTURY.*

By HENRY CARRINGTON BOLTON, Ph.D.

(Concluded from p. 232).

GREAT BRITAIN.

Society for Philosophical Experiments.

Founded in 1794 at London.

Publication.—Minutes of the Society for Philosophical Experiments, 1794.

Note.—A German translation of the *Minutes* was edited by Alex. Nic. Scherer and published at Halle in 1803.

Chemical Section of the British Association for the Advancement of Science.

Founded in 1831. This is, however, a corporate part of the British Association, and the papers read to the Section are published in the annual Reports of the British Association, 1831-1900. In 1900: President, W. H. Perkin, jun.; number of members not given.

Chemical Society of London.

Founded in 1841 at London. In 1900: President, T. E. Thorpe; honorary and foreign members, 33; members, 2300.

Publications.—Memoirs and Proceedings of the Chemical Society of London (1841-48); Quarterly Journal, 1849-62; Journal of the Chemical Society, 1863 1900.

Society of Public Analysts.

Founded in 1874 at London. In 1900: President, Walter W. Fisher; honorary members, 9; members, 260.

Publication.—Proceedings of the Society of Public Analysts, 1876; The Analyst, 1877-1900.

Institute of Chemistry of Great Britain and Ireland.

Founded in October, 1877, in London; incorporated, 1885. In 1900: President, John Millar Thomson; members, fellows, and associates, 1008 (resident members, 904); students, 118. Total, 1126.

Publications.—Proceedings, half-yearly, 1878 1900; Register, yearly, 1878-1900; Regulations, yearly, 1878-1900.

* Read at the Twenty-fifth Anniversary of the American Chemical Society, held in New York City, April 12—13, 1901. From advance proofs of the *Smithsonian Miscellaneous Collections*, 1901.

Society of Chemical Industry.

Founded in 1881 at London. In 1900: President, Charles F. Chandler; honorary member, 1 (John Glover); members, 3459.

Publication.—Journal of the Society of Chemical Industry, 1882-1900.

Note.—The Society has eight Sections—London, Liverpool, Manchester, Newcastle, New York, Nottingham, Scotland, and Yorkshire.

Society of Dyers and Colourists.

Founded in 1884 at Bradford. In 1900: President, H. Grandage; honorary members, 3; members, 553.

Publication.—Journal of the Society of Dyers and Colourists, 1884-1900.

Alembic Club.

Founded in 1889 at Edinburgh. This is a private club of only six members and has no president; the Secretary is Leonard Dobbin.

Publishes no journal, but has issued 15 Reprints of Chemical Monographs, &c., 1893-1900, and other works.

International Association of Leather-Trades Chemists.

Founded September, 1897, at London. In 1900: President, H. R. Procter; number of members, ——.

Publication.—Report of the Proceedings of the Conference of Leather-Trades Chemists, 1897.

ITALY.

Associazione chimico-farmaceutica fiorentina.

Founded in 1877 at Florence. In 1900: honorary members, 20; resident members, 100.

Publication.—L'Orosi, Bollettino di chimica, farmacia e scienze affini. Firenze, 1878-1900.

Società chimica di Milano.

Founded in February, 1895, at Milan. In 1900: President, Angelo Menozzi; resident members, 152; correspondents, 133.

Publication.—Annuario della Società chimica di Milano, 1896-1900.

Associazione chimico-industriale di Torino.

Founded June 25, 1899, at Turin. In 1900: President, Vittorio Sclopis; honorary members, 4; resident members, 103; correspondents, 87.

Publication.—La Chimica industriale, 1899-1900.

Società italiana dei chimici analisti.

Founded in 1893 at Pavia.

Publication.—Atti ufficiale delle Società italiana dei chimici analisti, 1893. This forms a pamphlet of 18 pp. only, and is perhaps a mere prospectus, as the Society ceased to exist before 1900.

JAPAN.

Chemical Society of Tokyo.

Founded April, 1878, at Tokyo. In 1900: President, Naokichi Matsui; number of members, 156; associates, 197.

Publication.—Tokyo Kagakkai Kaishi, 1880-1900.

Society of Chemical Industry of Japan.

Founded February, 1898, at Tokyo. In 1900: President, Takeaki Enomoto; honorary members, 7; members, 223; associates, 429.

Publication.—Kōgyō Kagaku Zasshi, 1898-1900.

RUSSIA.

Russkago Khimicheskago Obschtchestva (Russian Chemical Society).

Founded October 26, 1868. The Chairman of the first meeting was D. Mendeléeff. In 1900: President, F. F. Petrushevsky; members, 327.

Publications. — Zhurnal Russkago Khimicheskago Obshtchestva. St. Petersburg, 1869-72, 4 vols.

Continued as—

Zhurnal Russkago Khimicheskago Obshtchestva i Fisicheskago Obshtchestva, 1873-78. 6 vols.

Continued as—

Zhurnal Russkago Fisiko-Khimicheskago Obschtchestva, 1879-1900.

SOUTH AFRICA.

Chemical and Metallurgical Society of South Africa.

Founded May, 1894, at Johannesburg.

Publication. — Proceedings of the Chemical and Metallurgical Society of South Africa, 1894-1897

SWITZERLAND.

Société Chimique de Genève.

Founded February 10, 1878, at Geneva. In 1900: President, F. Kehrmann.

Publication.—The Minutes of the monthly meetings are published in Archives des sciences physiques et naturelles de Genève, and in the Chemiker Zeitung.

Verein schweizerischer analytischer Chemiker.

Founded March 12, 1887, at Zürich. In 1900: President, A. Bertschinger; number of members, 94.

Publication.—The organ of the Society is Schweizerische Wochenschrift für Chemie und Pharmacie, which was established under the title Schweizerische Zeitschrift für Pharmacie, 1856-62.

UNITED STATES OF AMERICA.

Chemical Society of Philadelphia.

Founded in 1792 at Philadelphia, under the presidency of James Woodhouse. Number of members unknown. The Society was in existence for more than ten years.

Publication.—Memoir on the Supply and Application of the Blowpipe (&c.), by Robert Hare, 1802.

Columbian Chemical Society of Philadelphia.

Founded August, 1811, at Philadelphia, under the presidency of James Cutbush; honorary members, 69; junior members, 13.

Publication.—Memoirs of the Columbian Chemical Society of Philadelphia. Vol. I., 1813.

Chemical Section of the American Association for the Advancement of Science.

A migratory organisation, founded in 1875 as a Sub-Section; it became Section C of the A. A. A. S. in 1882 at the second meeting in Montreal. In 1900: Chairman of the Section, Jas. Lewis Howe; members, 89; fellows, 181.

Publications.—The Proceedings of the A. A. A. S. has a division containing papers read before the Section of Chemistry.

American Chemical Society,

Founded April 20, 1876, in New York City. In 1900 President, William McMurtrie; honorary members, 10; members, 1546; associates, 123.

Publications.—Proceedings of the American Chemical Society, 1877-78; Journal of the American Chemical Society, 1879-1900.

Note.—In 1900 the Society had twelve Sections:—Rhode Island, Cincinnati, New York, Washington, Lehigh Valley, Chicago, Nebraska, North Carolina, Columbus, North Eastern, Philadelphia, and Michigan.

Association of Official Agricultural Chemists.

Founded September 8, 1884, at Philadelphia. In 1900: President, B. W. Kilgore; members, 350.

Publications.—Methods of Analyses, 1884-88; Proceedings, 1889-1900.

Note. — Conventions of the Official Agricultural Chemists had been held prior to 1884: in 1880, at Washington and Boston; in 1881, at Cincinnati; and in May, 1884, at Atlanta.

Chemical Society of Washington.

Founded at Washington in 1884. In 1893 became the Washington Section of the American Chemical Society, retaining also its name as above. In 1900: President, H. Carrington Bolton; members, 114.

Publication.—Bulletin of the Chemical Society of Washington, 1884-1892.

New England Association of Chemistry Teachers.

Founded February 19, 1898. Meetings are held in New England. In 1900: President, Rufus P. Williams; honorary members, 8; active members, 50; associates, 22.

Publications.—Circulars of Information and Reports, 1898-1900. Also Registers.

VICTORIA.

Society of Chemical Industry of Victoria.

Founded in 1900 under the Presidency of Orme Masson; membership, about 100.

ADDENDUM.

Société d'Arcueil.

Founded in 1807 at Arcueil. Dissolved in 1822. Members (at any one time), 12.

Publication.—Mémoires de physique et de chimie. Paris, 3 vols., 8vo. 1807-17.

Note.—This private organisation was founded by C. L. Berthollet; the meetings were held at his country house in Arcueil, near Paris. The membership included La Place, C. L. Berthollet and his son A. B. Berthollet, Biot, Gay Lussac, Humboldt, Thénard, Decandolle, Colle-Descotils, Berard, Chaptal, Dulong, Poisson, Malus.

The foregoing list does not include Academies of science nor Associations of general science (with a few exceptions); it does not embrace societies having for their object industries involving chemical processes in part, except the refining of sugar; nor does it include the numerous societies of brewers and of beer-making, among which may be named the following:—

Brau-Industrie Verein im Königreich Böhmen, founded at Prague in 1874, and publishing the Böhmische Bierbrauer.

Deutsche Brauerbund, founded at Nürnberg in 1861, and publishing the Allgemeine Hopfen-Zeitung.

Württembergische Brauerbund, founded at Waldsee in 1872, and publishing the Schwäbische Bierbrauer.

Badische Brauerbund, founded at Nürnberg in 1876, and publishing the Hopfenlaube.

Deutsche Braumeister Verein, founded at Berlin, 1887, and publishing the Deutsche Brau-Industrie.

Association générale des brasseurs Belges, founded at Brussels in 1874, and publishing Revue des Bieres.

County Brewers' Society, England, publishing since 1871 the Brewers' Guardian.

LONDON WATER SUPPLY.

Report on the Composition and Quality of Daily Samples of the Water Supplied to London for the Month Ending April 30th, 1902.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To Charles Perrin, Esq., M.Inst.C.E.,
Water Examiner, Metropolis Water Act, 1871.

London, May 9th, 1902.

Sir,—We submit herewith, at the request of the Directors, the results of our analyses of the 208 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from April 1st to April 30th inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 208 samples examined by us chemically during the month, all were found to be clear, bright, and well filtered.

The abnormal drought still continues; the amount of rain that has fallen at Oxford during the month is 1·21 inches, the average fall is 1·61 inches; this leaves a deficit of 0·40 inch, which, if added to the previous deficit for the year, brings it to 2·94 inches, or 42·2 per cent on the thirty-five years' average.

Our bacteriological examinations of 532 samples have given the results recorded in the following table; we have also examined 62 other samples, from special standpipes, wells, &c., making a total of 594 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 26 samples) ..	282
New River, filtered (mean of 132 samples) ..	9
Thames, unfiltered (mean of 26 samples) ..	10652
Thames-derived water, from the clear-water wells of eight Thames-derived supplies (mean of 289 samples)	31
Ditto ditto .. .. highest	496
Ditto ditto .. .. lowest	0
River Lea, unfiltered (mean of 26 samples) ..	545
River Lea, from the East London Company's clear-water wells (mean of 33 samples) ..	26

Of the 454 samples of impounded and filtered water supplied to London and examined bacteriologically during the month, 27 samples, or 5·9 per cent, were sterile. Thirteen samples contained more than 150 microbes, while thirty-one samples, or 7·0 per cent, contained more than 100 microbes per c.c. The mean number of microbes in the thirty-one excess samples was 181, against a corresponding mean of 166 in seventeen excess samples during March. These excess samples were by no means equally distributed among the various companies.

Taken as a whole, the Metropolitan water supply more than maintains the high standard of purity which has characterised it during the previous months of this year.

We are, Sir,
Your obedient Servants,

William Crookes.
James Dewar.

A NEW INVESTIGATION CONCERNING THE ATOMIC WEIGHT OF URANIUM.*

By THEODORE WILLIAM RICHARDS
and
BENJAMIN SHORES MERIGOLD.

(Concluded from p. 230).

It is worth while to enquire whether or not the method may conceal some source of constant error beyond the reach of the experimental precautions detailed above. Such an error could hardly have occurred during the analysis; for every step of this procedure was verified by confirmatory evidence. If a flaw existed, it must have been in the purity of the original substance. Since the observed atomic weight is lower than the former results, it is important to examine into only those possible irregularities which could have had the effect of lowering the apparent value.

The probable impurities tending to lower the atomic weight are, first, sodic bromide; second, hydrobromic acid; third, free bromine; fourth, uranic pentabromide; and fifth, an unknown metal with a lesser equivalent. The first impurity was found to be present, its amount was determined, and a suitable correction was applied. The second could not have been formed during the sublimation of the uranous bromide, because compounds of hydrogen were scrupulously excluded. If formed by the action of water after the sublimation, the atomic weight would have appeared too high—for moist uranous bromide emits hydrobromic acid instead of absorbing it. The third impurity, free bromine, could hardly have been imprisoned or absorbed by the sharply crystalline salt to any appreciable extent, since the concentration of the bromine vapour in the issuing gases was but small.

The evidence in regard to the absence of pentabromide is fairly conclusive, although somewhat indirect. All attempts by many investigators to form this compound have failed, in spite of the recognised existence of the corresponding chlorine compound. It seemed possible, however, that while this compound is not formed at high temperatures, lower temperatures might permit the addition of the extra bromine. Accordingly the preparations used in Analyses 7, 8, 10, and 11, were cooled in a current of dilute bromine vapour, instead of in pure nitrogen. The presence of a comparatively small amount of pentabromide would make a very decided difference in the quantity of bromine found. Hence the essential agreement of the average result of these analyses, 238·50, with the average result of all the others, 238·52, is good evidence of the absence of uranium pentabromide.

With regard to the fifth possible impurity nothing can be said except to point out the many operations involved in the purifications. These seem to point toward probable purity; but it is nevertheless to be regretted that lack of time prevented the analysis of many different fractions of material, prepared in varying ways.

The presence of oxybromide would, of course, cause low bromine analyses, and too high an apparent atomic weight. Therefore this possible cause of error need not be considered, even if the oxybromide had ever been made in the absence of water. In the light of all these considerations, there would seem to be no good reason to question the purity of our bromide.

On comparing the result of this investigation, 238·53, with that of Zimmermann's, 239·59 (the only previous work worthy of serious consideration), the difference of over a unit seems at first to be one of great magnitude. The percentage difference (0·45 per cent) is, however, smaller than many a difference which often has been passed by unheeded in small atomic weights, such as those of magnesium or aluminum. This point illustrates the difficulty of obtaining results with high atomic weights which can satisfy the cursory reader.

Nevertheless, such a difference is far too great to pass unchallenged. It seems highly probable that the greater part of it is due to the previously discussed sources of inaccuracy in Zimmermann's method—especially to the difficulty of wholly re-oxidising the lower oxide. The failure to oxidise half a per cent of the uranous oxide, involving an error in the weight of only 0·017 per cent of the total weight of the substance, would account for the discrepancy.

Hence it seems not unlikely that the atomic weight of uranium is really as low as 238·53. Nevertheless, the question cannot be looked upon as conclusively settled. Certainty can be obtained only by the application of a new method, radically different from the two just compared. Our experience of nearly four years of varied work seems to indicate that the search for such method will not be an easy one. The many degrees of quantivalence of uranium and the unsuitable properties of its compounds combine to render the problem one of unusual difficulty. When face to face with a problem of this kind one cannot but admire the wisdom of Stas in selecting chiefly univalent elements with powerful affinities in order to prove the constancy of the atomic weights.

The result of our analyses of uranous bromide may be summed up in the following words:—If oxygen is taken as 16·000, and bromine as 79·955, the atomic weight of uranium appears to be not far from 238·53.

* Contributions from the Chemical Laboratory of Harvard College. From the *Proceedings of the American Academy of Arts and Sciences*, xxxvii., No. 14.

NOTICES OF BOOKS.

A Text-book of Inorganic Chemistry. By A. F. Holleman. Rendered into English by Hermon C. Cooper, with the co-operation of the Author. New York: John Wiley and Sons. London: Chapman and Hall, Lim. 1902. 8vo. viii.-458 pp. Ill.

This superior text-book made its first appearance in 1898 at Groningen, where the author holds a Chair in the University. Two years later a German translation was published, and has met with great success, having been adopted at the Universities of Würzburg, Freiburg, Munich, Breslau, Göttingen, Zürich, Vienna, St. Petersburg, Tokyo, and elsewhere. English and American students will welcome this translation, which has been carefully made by Dr. Cooper.

The distinctive features making this book a decided advance on its predecessors are tersely stated by the German reviewer of the edition that appeared in 1900. He wrote:—"The work combines the new achievements of physical chemistry with the mass of long-established facts of inorganic chemistry so as to form a unified whole." It will not be necessary hereafter for students to get acquainted with the common phenomena of elementary chemistry by the perusal of one book written on the old plan, and then to take up the independent study of the laws of physical chemistry (notably those established by van 't Hoff, Ostwald, and Arrhenius, and their disciples), as set forth in some other manual devoted to that subject. He will find that Holleman combines all these points in one volume. Sections on osmotic pressure and its relation to molecular weights, on dissociation, electrolytic dissociation, thermo-chemistry, methods of determining atomic weights, the periodic system, and electro-chemistry, all in their most modern aspects, are introduced at intervals among purely descriptive and didactic pages, so naturally that they seem to make a part of the science, and not independent theories invented for the amusement of their authors and their friends.

At the same time the descriptive matter is most judiciously selected, and the theoretical views are introduced at such points that they can be illustrated and explained by the matters under consideration, thus presenting a harmonious view of the science. The theories

are treated on a mathematical basis, but the calculus is used very sparingly.

Without prejudice, the names of persons to whom credit is due are mentioned in connection with their work. The Americans, Gibbs, Clarke, Morley, Saunders, receive notice alongside of the Europeans, van 't Hoff, Arrhenius, Ostwald, Nernst, Berthelot, and others.

The translation is satisfactory and generally free from ambiguity. On page 289, however, near the bottom, the discovery of germanium is said to have been made by the chemist who named an unknown element "ekasilicon," owing to the misuse of a pronoun. The proof-reading shows great care, but on page 275, in the table, for "Sp. G." read "Sp. H." The expression "spectrum analysis" is preferred by the reviewer to "spectral analysis."

The author is to be congratulated on having written a capital text-book, so fresh in its treatment, so entirely up-to-date in matter, so accurate, and so utilitarian as to place it far above any work having the same objects in view that has appeared during the last twenty-five years. If we are not mistaken in our estimate, Holleman's "Inorganic Chemistry" will prove an epoch-making text-book.

There is a good index.

The same publishers promise a translation of Holleman's "Organic Chemistry," prepared by Drs. Walker and Mott, of Derby. H. C. B.

August Wilhelm von Hofmann; Ein Lebensbild. By Jacob Volhard and Emil Fischer. Berlin: R. Friedlander und Sohn. 1902.

The work of preparing a biography of Hofmann was originally entrusted by the German Chemical Society to Ferdinand Tiemann and Emil Fischer. On the premature death of the former, however, Jacob Volhard undertook his portion of the task. This book, the outcome of the joint labours of the pupil and successor of Hofmann, is divided into two parts. The first is purely personal and social, and gives us a delightful picture of Hofmann, the man; many of his letters to members of his family are inserted in full, and it is easy to trace in them the characteristics which marked him out as a most successful author. The simple, ingenuous, and yet acute nature of the great chemist is laid bare before us, his domestic joys and sorrows, and his dealings with the College of Chemistry, which in its early years encountered so many difficulties from which it finally emerged under Hofmann's skilful guidance. Some of the letters written from Osborne, whither he went by Royal Command to lecture before the Queen after the death of the Prince Consort, display an extraordinarily vivid sense of humour, and it is difficult to resist lingering over this portion of the book, which is fascinating reading for all who can appreciate a good biography.

The second part, written by Emil Fischer, is devoted to Hofmann's scientific work. Here, owing to his enormous activity, the summariser's task has been by no means easy. His extensive work upon aniline, and organic nitrogen compounds generally, his influence on the coal-tar colour industry, his work on the aliphatic mustard oils, to mention only a few examples, are all described as fully as space permits, and all will agree with the author that he was the greatest of Liebig's pupils, and that his name may well be coupled with those of the heroes of German Chemistry. The whole book gives us a vivid picture of a delightful man, an inspiring teacher, and an extraordinarily able and successful investigator.

Beiträge zur Chemischen Physiologie und Pathologie. Edited by Franz Hofmeister. Band II., Heft 4. Braunschweig: Friedrich Viewig und Sohn. 1902.

This short number of the *Beiträge* includes a paper by Dr. Ernst Fuld on the compounds of albuminous bodies with metaphosphoric acid. Without committing himself to a definite statement, the author conjectures that, for serum albumen, from the percentage of phosphorus in the metaphosphoric acid compounds, a molecular weight of 5590 may be assumed for the phosphate and 5100 for the pure albumen. The latter number agrees closely with the lowest obtained from the percentage of sulphur. Another paper, occupying a considerable part of the number, deals with the curdling effect of rennet upon milk, examined both from a theoretical and practical standpoint. In a short communication on a modification of Kjeldahl's method of determining nitrogen in organic substances, the use of sodium thiosulphate is advocated to decompose the amido-mercury salts formed on addition of mercuric oxide. Commercial thiosulphate is said to be free from nitrogen, and has the advantage of being able to be used in the solid state, whereas alkali sulphides have to be employed in aqueous solution, thus inconveniently increasing the volume of liquid to be distilled, and lengthening the process.

Handbook of Technical Gas Analysis. By Clemens Winkler, Ph.D., Professor of Chemistry at the Freiberg Mining Academy. Second English Edition. Translated from the Third, greatly enlarged, German Edition, with some Additions, by George Lunge, Ph.D. London: Gurney and Jackson. 1902. Pp. 190.

The third German edition of this book was published in 1901, and as the first English edition was just exhausted, the proof-sheets of the former were kindly lent to the translator for the preparation of this second English edition. The book has been considerably enlarged, the number of pages having been increased by one-half, though the scope of the work remains the same. Even now all the processes and apparatus proposed for gas analysis are not included, but only the most important of them, including the valuable additions to technical analysis made by Prof. Hempel.

Gas analysis is usually effected by measuring and not by weighing, and for the most part the results are given in parts per cent, or per 10,000 by volume, which volume must be corrected for pressure and temperature.

The analytical process followed in the examination of gases generally consists in transforming one constituent after the other into a compound of a different form. From the contraction of volume thus produced, the volume of each special constituent can be deduced, directly or indirectly. This can be done in three principal ways:—1. By direct absorption. 2. By combustion. 3. By combustion and subsequent absorption of the products. And, according to how the process is conducted, gases can be estimated either by direct measuring, by titration, or by weighing.

Chapters I. and II. are on taking samples of gases, and general remarks on their measurement; and Chapter III., which forms the principal part of the book, deals with the apparatus and methods for carrying out the analysis of gases. This chapter is divided into three principal subdivisions on—the estimation of solid and liquid admixtures, the estimation of gases by absorption, and the estimation of gases by combustion; and in every case the method and procedure are fully and clearly described, while an important feature of the book is the illustrations, which are numerous and excellent. The appendix contains a number of tables indispensable for working out results, and, finally, there is an index.

Les Combustibles; Solides, Liquides, Gazeux. ("Fuels; Solid, Liquid, and Gaseous"). By H. J. Phillips, F.I.C., F.C.S. Translated from the Third English Edition by J. Rossert. Paris: Gautier-Villars. 1902. Pp. 165.

Fuels are at the base of almost all industries, whether by transforming their heat of combustion into mechanical energy—as in steam, petroleum, or gas engines—or for transforming natural products into commercial materials,

as in metallurgy. In every case it is important to know the exact value of the fuel used; and this value depends not only on its calorific power, but also on its chemical composition. The presence of sulphur, phosphorus, &c., in fuels makes them useless for certain purposes, so an exact analysis is of importance.

The scope of this book is to give the simplest methods for the analysis of solid, liquid, and gaseous fuels, as well as the methods for the determination of their calorific power.

At the end of the book is a series of tables giving the composition and calorific power of all kinds of commercial fuels, such as coal, coke, petroleum, and furnace gases from both regenerative and blast furnaces.

The references in the index do not seem to be as simple as they might. Instead of giving the number of the page, the chapter and number of the paragraph are given. Thus:—Anthracite, VII., 4; VII., 24; Hydrogen (estimation of), II., 22; Oxide of iron, VI., 5; &c.

CORRESPONDENCE.

SCHRÖTTER'S CARBONIC ACID APPARATUS.

To the Editor of the Chemical News.

SIR,—I have recently performed a number of analyses of baking-powder, and as I found the usual form of Schrötter's CO_2 apparatus extremely difficult to clean, and more especially to dry, I have designed a modification which may be useful to some of your readers.

The ordinary tap-funnel and drying tubes of a Schrötter are sealed into a tube, which is then accurately ground into a weighing bottle in place of the stopper. If the long tube form of tap-funnel is used, that joint must be an inserted joint. The ground joint must be well made. This apparatus is, of course, very easy to clean and dry, while it is as accurate as the older form.

My model was made by Messrs. C. E. Muller and Co., of High Holborn, and I believe that they intend to stock it.—I am, &c.,

C. E. KENNETH MEES.

The Chemical Laboratory,
University College, W.C., May 13, 1902.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxiv., No. 17, April 28, 1902.

Researches on Electric Piles formed by the Reciprocal Action of Oxidising and Reducing Liquids. Common Solvents. Action of Acids on Bases.—M. Berthelot.—The author performs a certain number of synthetic experiments, in order to show the order of chemical reaction susceptible of being produced under normal conditions in the midst of the tissues of living beings. He succeeds in liberating the acids, even the most energetic of them, such as hydrochloric and sulphuric, by electrolysis—acids whose existence is established either amongst the secretions of the stomach or on the surface of the organs.

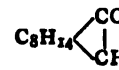
Composition of Hydrate of Chlorine.—M. de Forcrand.—The composition of hydrate of chlorine has been much discussed. Faraday prepared this substance by desiccation of the crystals in the cold, and published a number of analyses, of which the mean gives as a formula Cl_2+11H_2O. M. Roozeboom, using the same method, found 28·1 to 31·9 per cent of chlorine, and he believed the hydrate to have the constitution Cl_2+6H_2O. By two general methods, already described by the author, he finally solves the question of the composition, and finds it to be Cl_2+7H_2O.

Some Derivatives of Oxyisopropylphosphinic Acid.—C. Marie.—Oxyisopropylphosphinic acid gives two series of salts, the neutral salts and the acid salts. He prepared the following:—*Sodium salts:* (*a*) neutral salt — $PO_3HNa_2(C_3H_6O),5H_2O$; (*b*) acid salt — $PO_3H_2Na(C_6H_6O),6H_2O$. *Lead salt:* $PO_3HPb(C_3H_6O)$. *Copper salts:* (*a*) acid salt—$PO_3H_2\frac{Cu}{2}C_3H_6O$; (*b*) neutral salt—$PO_3HCu,C_3H_6O,H_2O$. *Silver salt:* PO_3HAg_2,C_3H_6O

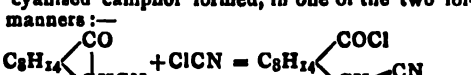
Bulletin de la Société Chimique de Paris.
Series 3, Vol. xxv., Nos. 22.

Integral Method for the Destruction of Organic Matters, applicable to the Search for Mineral Poisons, especially Arsenic and Antimony.—G. Denigès.—Already inserted in full (vol. lxxxv., p. 26).

Particular Action of Chloride of Cyanogen on Soda-Camphor.—H. Duval.—While attempting to prepare cyanised camphor by the action of chloride of cyanogen on soda-camphor in solution in toluene, the author obtained a body which could not be identified either with the sought for product or any other known substance. After the passage of the chloride of cyanogen, the toluenic solution was extracted with weak soda. This latter solution, treated with an acid, gave a small quantity of a yellow coloured amorphous body; the return, however, was only 3 per cent. To crystallise this product, ether, benzene, and water were successively tried. The ether left a gummy mass; in benzene it crystallised in deep yellow-coloured flakes; with water almost colourless needles were obtained by the first crystallisation. Analysis showed that this substance contained nothing but carbon, hydrogen, and nitrogen. It is soluble in alkalis and easily displaces carbonic acid in the carbonates when heated; it may be, therefore, a carboxylised compound. As for its formula, if camphor be taken as—

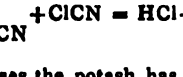


we can see that the new body ought to contain two atoms of nitrogen and one carboxylised function; on the other hand, the method used warrants the supposition that a fresh molecule of the halogenised derivative has reacted on the cyanised camphor formed, in one of the two following manners:—



Or,

and that in both cases the potash has caused, by substitution or by scission, the formation of the substance—



The figures found by analysis give us:—

	Found.	Calculated for $C_8H_{14}<^{CO_2H}_{CH<^{CN}_{CN}}$
N	13·04	12·72
C	65·24	65·45
H	7·34	7·22
O (by diff.)	14·38	14·54

The Mechanism of the Etherification of Plants.—MM. Charabot and A. Hebert.—Already noticed.

The Agricultural Value of the Nitrogen in the Black Materi. l of the Phosphates from the Pyrenees.—J. Joffre.——Already inserted in full.

MISCELLANEOUS.

The Andrew Carnegie Research Scholarships.—The Andrew Carnegie Gold Medal for 1902 has been awarded by the Council of the Iron and Steel Institute to Dr. J. A. Mathews, of New York, for the research carried out by him as a holder of an Andrew Carnegie Research Scholarship during the past year. The medal has been designed by Mr. G. W. de Saulles, of the Royal Mint. The first recipient, Dr. Mathews, has previously received a Fellowship for the encouragement of scientific research from Columbia College, New York, where he has been working under the guidance of Professor H. M. Howe. For the present year the Council of the Iron and Steel Institute has awarded six Andrew Carnegie Research Scholarships each of the value of £100. The following are some particulars of the careers of the successful candidates:—

Octave Boudouard, of Paris, aged 30, has already published thirty-two original memoirs, and, in conjunction with Professor Le Chatelier, four books. He is Assistant Professor of Chemistry at the College of France, and has previously received in France bronze and silver medals for research.

William Campbell, of New York, aged 25, has published papers for the Institution of Mechanical Engineers, the American Chemical Society, and the Franklin Institute. He studied at the Durham College of Science, and was awarded an 1851 Royal Exhibition Scholarship to the Royal School of Mines, London. He is now working under Professor H. M Howe at Columbia College.

Alfred Campion, of Cooper's Hill, aged 27, is a member of the Iron and Steel Institute, who has had practical experience with the Steel Company of Scotland, and has previously written papers for Glasgow Societies.

Percy Longmuir, of Manchester, aged 25, was a student of the Sheffield University College. He has had experience in foundry work, and has written several papers on that subject.

Ernest Schott, of Berlin, aged 26, studied under Professor Ledebur, of Freiberg, and is Assistant in the Royal Testing Institution at Charlottenburg. He has published a number of original memoirs.

Frederick Henry Wigham, of Wakefield, aged 32, is a member of the Iron and Steel Institute. He is Steel Works Manager to Messrs. George Cradock, and Co., and communicated, in conjunction with Mr. Stead, a paper on steel for wire-making to the Iron and Steel Institute last year.

The Substitutions of Elements of the Sulphur-selenium-tellurium Group.—J. Krafft and O. Steiner.—If we heat selenide of phenyl, $(C_6H_5)_2Se$, in an open beaker with its equivalent quantity of sulphur, going nearly to boiling-point, the selenium is almost entirely displaced, and we obtain sulphide of phenyl, $(C_6H_5)_2S$. We can also operate at 300° in a sealed tube filled with CO_2; the selenium is deposited in the metallic state, and if we take up with ether, to dissolve the sulphide formed, we find that 95 per cent of the sulphur has displaced the corresponding quantity of selenium. Similar results are obtained by the action of sulphur on telluride of phenyl, $(C_6H_5)_2Te$, but it is necessary to operate at 280° in a sealed tube; after heating for ten hours 58 per cent of the tellurium is displaced, bisulphide of phenyl, $(C_6H_5)_2S_2$, rather than monosulphide, is formed. As for oxygen, it does not act on sulphide of phenyl, but inversely sulphur acts slightly on oxide of phenyl, $(C_6H_5)_2O$; after heating for fifteen hours at 340°, a small amount of sulphide is formed, and many products of alteration. Sulphur heated to 110° in a sealed tube with selenious anhydride gives a selenium of the black variety, and sulphurous anhydride, which is liquefied. When heated to 55° with crystallised selenic acid, the sulphur is oxidised to the state of sulphuric acid, and causes the formation of selenium and selenious acid. Inversely, chloride of sulphur is decomposed by selenium and tellurium; the chloride of selenium, $SeCl_2$, decomposes on distillation into $SeCl_4$ and Se. In the case of tellurium, the chloride formed by the following equation can be isolated by distillation without decomposition:—$S_2Cl_2 + Te = TeCl_2 + 2S$. When sulphobenzide, $C_6H_5.SO_2.C_6H_5$, is distilled with selenium or tellurium, we obtain the selenide or telluride of phenyl, and at the same time SO_2.—*Berichte*, vol. xxxiv., p. 560.

MEETINGS FOR THE WEEK.

Tuesday, 27th.—Royal Institution, 3. "The Laws of Heredity, with special reference to Man," by Prof. Carl Pearson, M.A., F.R.S.

—— Society of Arts, 8. "Pageantry and the Masque," by Miss May Morris.

Wednesday, 28th.—Chemical, 5.30. "Taxin," by T. E. Thorpe and G. Stubbs. "Soil Samples" and "Some Excessively Saline Indian Well Waters," by J. W. Leather.

Thursday, 29th.—Royal Institution, 3. "Contemporary British Sculpture," by M. H. Spielmann.

—— Society of Arts, 4.30. "Western Australia—its Progress and Resources," by the Hon. H. W. Venn.

Friday, 30th.—Royal Institution, 9. "The Progress of Electric Space Telegraphy," by G. Marconi, M.Inst.E.E.

Saturday, 31st.—Royal Institution, 3. "The Development of the English Drama—II., The Drama of the Middle Ages," by Prof. Brander Matthews, Litt.D., D.C.L.

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INTRODUCTION to the STUDY of CHEMICAL PHILOSOPHY; the Principles of Theoretical and Systematic Chemistry. By WILLIAM A. TILDEN, D.Sc. Lond., Sc.D. Dub., F.R.S., Professor of Chemistry in the Royal College of Science, London. TENTH EDITION, Fourteenth Impression (1901), completely Revised and Rearranged. Fcap. 8vo, 5s. With Answers to Problems, 5s. 6d.

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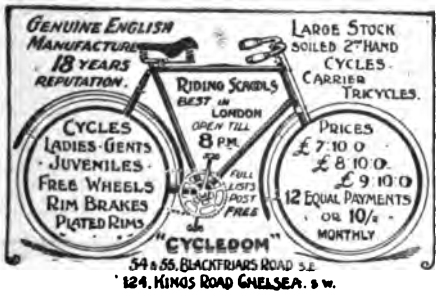
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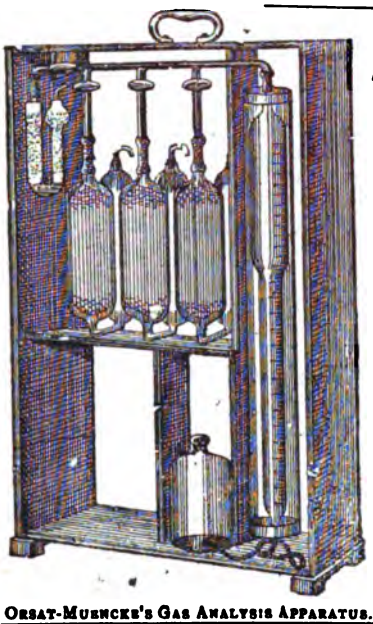

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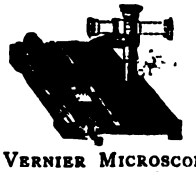

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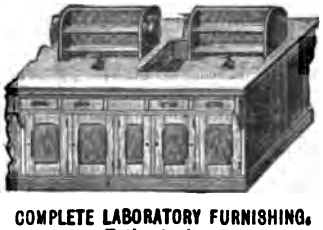

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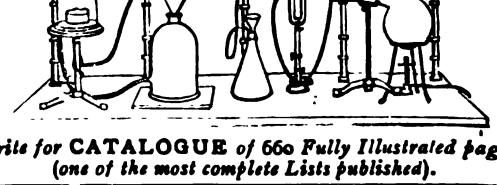

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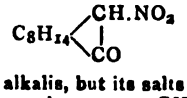
VOL. LXXXV., No. 2218.

THE CONDITIONS DETERMINATIVE OF CHEMICAL CHANGE AND OF ELECTRICAL CONDUCTION IN GASES, AND ON THE PHENOMENA OF LUMINOSITY.*

By HENRY E. ARMSTRONG, V.P.R.S.

(Concluded from p. 243).

IN a case like that of radium, it would seem that energy of low period suffices to bring about the change the reversal of which subsequently gives rise to the luminous effect and radio-activity. The chemist is tempted to contrast the behaviour of radium with that of a substance undergoing change into an isodynamic form—such as nitro-camphor, for example. Nitro-camphor—



dissolves readily in alkalis, but its salts are derived from a pseudonitro-camphor, the group $CH.NO_2$ undergoing alteration into the group C(NO.OH). When liberated from its salts, the pseudo-form at once, in great part, reverts to the normal form; on the other hand, when solid nitro-camphor, which apparently consists almost entirely of the normal form, is dissolved in a liquid, it in part gradually undergoes conversion into the pseudo-form. A point of equilibrium is eventually reached when from 7 to 10 per cent of the pseudo-form is present (compare Lowry, *Chem. Soc. Trans.*, 1898, p. 966). The change proceeds at different rates in different solvents, and is undoubtedly conditioned by some catalyst; in other words, either form of the *pure* substance would be stable. It will be apparent that the change is one which does not affect the molecule as a whole, but merely an isolated region in it. If the energy set free in the formation of the dominant form were of sufficiently high period, nitro-camphor might appear luminous and even be radio-active. The argument is of interest, as it serves to suggest that radio-activity may not be the isolated phenomenon we at present suppose, but a concomitant of many chemical changes.

[From this point of view, it appeared to be desirable to examine substances such as saccharin (orthobenzoic sulphuride) and cane-sugar, which glow in a very remarkable manner when broken up. Mr. E. W. Lewis has kindly made a number of experiments for me, but without obtaining positive results; although an intense photographic impression of the flash is easily obtained by merely crushing crystals of saccharin or sugar on a glass plate held above a sensitive film, crystals placed on a film are without effect, even when left in contact with it during several days. It will, however, be desirable to apply the far more delicate electroscopic test to such substances under various conditions. It so happens that the property referred to was first noticed in the case of saccharin in my laboratory (in 1895) by Mr. Jackson Pope (*Chem. Soc. Trans.*, 1895, p. 985), and in March last I applied to this gentleman for a specimen of the substance he had prepared in 1895. He was able to send me crystals, but informed me that those he had tested were inactive. We found this to be true of the remainder, at most a very feeble glow being observed on crushing them; but active crystals were obtained on re-crystallising the inactive material from acetone. Old specimens of sugar crystals appear to be as active as new ones.—*Note, added May 2*].

A class of effects attendant on radio-activity, and which have an important bearing on the question previously considered, are those attributed to emanations from radio-active substances. Sir William Crookes has brought the subject prominently under notice in his recent communication. The behaviour of such emanations is strikingly similar to that of ordinary gross matter; indeed, it is difficult to resist the conclusion that such is their character, and that they are but secondary products engendered by radio activity.

[Professor Rutherford's recent observations on the "emanation" from thorium compounds are very difficult to interpret. The assumption that Mr. Soddy and he are inclined to make (*Chem. Soc. Trans.*, 1902, p. 321), that the emanation is allied in properties to the elements of the argon group, is almost a contradiction in terms. Such a constituent would presumably be an inert substance, and if not removed by the drastic treatment to which they submitted their material without permanently affecting its radiation power, it would scarcely then escape spontaneously on mere exposure of the solid. It would seem to be far more probable that the "emanation" is a secondary manipulation, in some way conditioned by the "straight-line radiation."

There is a feature in Dr. Russell's experiments which has always struck me as remarkable, assuming that the photographic effects he has obtained are due to hydrogen peroxide, viz., the absolute sharpness of the images formed by a scratched zinc plate, even when this is placed at a relatively considerable distance above the sensitive film. If the emanation passed by mere diffusion from the active surface, a more or less blurred image might be expected to form. It would almost seem that the molecules are projected in straight lines—in other words, that they may be electrically charged. If so, a distinction should perhaps be drawn between a merely vapourised substance and a "nascent" substance.—*Note, added May 2*].

The production of hydrogen peroxide under such an influence at once presents itself not merely as a possibility, but practically as a necessity. There is every reason to suppose that hydrogen peroxide is a necessary product of oxidation by ordinary oxygen whatever the substance oxidised; but the conditions under which it is produced are usually such as to engender its own immediate decomposition—so that it either escapes observation or is met with only in minute quantity. The oxidation of zinc may be taken as an example. The oxidation necessarily takes place in a circuit comprising (*a*) the impure metal—which because it is impure furnishes both negative and positive electrode—(*b*) conducting (impure or dirty) water, and (*c*) oxygen, of which the last acts as depolariser. The water is electrolysed, its oxygen going to the metal; at the same time, the hydrogen from the water becomes associated with the depolarising oxygen *molecule*, forming hydrogen peroxide, H_2O_2: the products, in fact, are zinc oxide *and hydrogen peroxide* in equivalent quantities, not merely zinc oxide as is taught by the text-books.

It does not seem to me that Sir William Crookes has disproved the possibility that the effects may be due to hydrogen peroxide by his experiment in which one limb of a U-tube passed through the cork in a bottle containing a solution of the peroxide, the open end of the other limb being put close to a sensitive film, the result being that no photographic effect was observed in seventy-two hours, although a strong effect was produced when the film was kept over the mouth of the bottle during only twenty-four hours. The peroxide molecules would undoubtedly tend towards the walls of the tube, especially at the bend; but collision therewith would probably have fatal effects, as the alkaline surface of the glass would promote their destruction.

The argument may be applied to recent researches on the so called ionisation of air and other gases. If this term implied the recognition of nothing more than the power of conducting, little difficulty would arise. Faraday, with characteristic and truly scientific caution, used the

* A Paper read before the Royal Society, May 1, 1902.

word *ions* simply "to express those bodies which can pass to the electrodes." Unfortunately, of late years the signification of the word has been entirely altered: ionisation now connotes a schism—a process of molecular suicide; and the ions are looked upon as discrete particles enjoying separate existence, as the electrically charged, wandering elements of molecular disruption. Before we assume that such ions are present in air under ordinary conditions, and that they alone can condition conductivity, it behoves us to examine carefully into the nature of the evidence which is relied on in proof of their presence. It appears to me that much of the work done in this field is open to criticism. Thus, in Mr. C. T. R. Wilson's experiments (*Roy. Soc. Proc.*, 1901, vol. lxviii., p. 151; vol. lxix., p. 277), even the minute amount of chemical change occurring at the surface of the sulphur bead attaching the gold leaf of the electroscope to its support, may have sufficed to render the atmosphere within the flask conducting. And in the later experiments of the same observer, the phosphoric anhydride used as drying agent may have contributed an acid impurity. Sir William Crookes has recently given an illustration of the manner in which impurities may be derived from phosphoric anhydride. By heating it strongly *in vacuo*, he got rid of these to such an extent that they were no longer recognisable by the spectroscope; but it cannot be supposed that even such treatment suffices to render the anhydride innocuous, bearing in mind the extraordinary delicacy of the electroscopic test.

I venture to think that until the phenomena of conductivity presented by gases have been studied not merely with the same, but even with far greater, care than has been devoted to the study of those attending gross chemical changes in gases, it is premature to conclude that gases undergo ionisation—using the word in its modern sense. I also venture to think that the question whether mere molecules cannot form conducting systems has not yet received in any way the attention it deserves from those engaged in these inquiries.

HISTORY OF THE INVENTION OF INCANDESCENT GAS-LIGHTING.

By AUER VON WELSBACH.

In the year 1880 I was occupied with the chemistry of the rare earths. The remarkable phenomena exhibited by some of these earths, when heated to a high temperature by a flame, excited my interest to a great extent. One body in particular, called erbia by chemists, behaved in an altogether singular manner. It did not become luminous like other bodies and give off white or yellow light, but its radiations were green. Science has not yet been able to give any explanation of this remarkable phenomenon. This fact, which was interesting and astonishing to the highest degree, may be compared with the observations recently made on the radiations from polonium, for example.

I refer to this phenomenon because it interested me very greatly, and was, so to speak, the starting-point of my discovery. The spectrum given by this earth is not continuous; it is a band spectrum such as is given by but few solid bodies.

The examination of the light emitted by the rare earths raised to incandescence was very important in the study of these compounds. But the little beads easily obtained on platinum wires are not suitable for the production of very luminous spectra. I therefore arranged the earths in the flame in such a manner that the emission of light should be much more intense. It occurred to me, quite by chance, to soak cotton with the solutions of the salts of these earths, and then to calcine them. It seemed probable that this experiment might not succeed, and that the skeleton formed by the earths, after the combustion of the cotton, would not have sufficient coherence. However, it was a success; the earths retained the shape of the cotton. Shortly afterwards, when I visited my old master, Bunsen, at Heidelberg, and explained to him how I had manipulated these compounds, he shook his head and said that it seemed impossible to him that a coherent mass of oxide could be obtained in that manner. I still remember with pleasure the astonishment he evinced when I was able to show him how I obtained my mantles.

During the course of my experiments to give these earths a structure appropriate for their luminous emissions, I came across one substance of which the very considerable emissive power was then but little known, for the reason, as I have stated, that these bodies had only been examined in the form of small beads. This compound was oxide of lanthanum, and it is this substance that gave me the idea of utilising the rare earths for the production of light on the large scale.

The mantle of oxide of lanthanum seemed perfect, but in it I found my first disappointment. I was away from the laboratory for several days, but had left the mantle carefully locked up; when I returned it was reduced to powder. I repeated the experiment with the same result. Oxide of lanthanum was always reduced to fine powder. My first joy was of short duration.

Then I began to think whether I could make oxide of lanthanum, whose emissive properties I was so absolutely certain of, more stable by mixing it with a compound which would attract the moisture and the carbonic acid of the air less easily. I experimented first on magnesia. I was already aware of the importance of the extreme division and the intimate mixture of the material to be calcined. The pulverisation ought to be integral, and I named these mixtures molecular mixtures. When we mix these bodies in this manner and calcine them, their properties undergo important modifications. White magnesia and white oxide of lanthanum, on being calcined, give a substance of a deep brown colour, and the properties of the compounds can be no longer recognised in the product. The first satisfactory compound for incandescent lighting with gas was formed of the oxides of lanthanum and magnesium. It did not fall to powder in the air after calcination, and it gave a beautiful light; however, its useful effect was not yet very great, it was just about that of a Siemens regenerating burner. But it had one great fault: after seventy or eighty hours of heating it became translucent and vitreous, losing its porosity. Thus, finally, this second attempt did not succeed.

I studied this question anew, from the chemical point of view; it was evident that magnesia did not resist fire sufficiently, that it could not undergo a prolonged calcination without being considerably modified. So I turned my attention to mixtures having oxide of zirconium as a basis. The results obtained were better. This mixture gave a steady light, and its life in the flame reached some hundreds of hours; this success gave me further encouragement, and at the same time I made experiments with oxide of thorium, and was surprised at the intense increase in luminosity of the mixtures containing this substance. These experiments showed that certain molecular mixtures of oxides constituted alloys, so to speak, which emitted a particularly intense and constant light when placed round a flame in the form of a fine net-work.

I already looked upon it as a certain fact that a new system of lighting could be founded on these phenomena, more economical and advantageous than the use of gas in the form of ordinary light-giving flames.

It must, however, be remembered—and this is the key to the invention of gas-lighting by incandescence—that the question was not to find a process by which an infusible compound could be given a definite shape. This invention is founded, above all, on the fact, proved by numerous experiments, that molecular mixtures of certain oxides are possessed of properties which cannot be deduced from those of their constituents,

Encouraged by all these results, and sustained by my experiments, I was bold enough to look upon the efforts made for the previous ten years to augment the lighting power of the gas-flame as useless. It is, in fact, evident that, the emissive power of these compounds being double or treble that of the simple flame with an equal consumption of gas, it is more economical to give up the use of a luminous flame. It is far better to use a non-luminous very hot flame to raise these new bodies to incandescence.

I now come to some well-known facts. I gave a small demonstration to the Viennese press in the laboratory of M. Lieben, and the reports, which were for the most part favourable, announced my invention to the public. While some looked upon this invention with approval, others affected a good deal of scepticism, or made fun of it. I am acquainted with a well-known gas engineer who was always ready to bet with any one he met, that less than 1000 burners a year would be installed in any town. Another one, when asked to interest himself in the new invention, said:—"In my works we only take notice of serious ideas."

These answers did not affect my confidence. Incandescence by gas was welcomed, and gradually gave rise to a certain amount of business. I was obliged to leave this subject for some time, my attention being occupied by contracts and other matters. For the purpose of continuing this work I found a very distinguished man who rapidly set about the manufacture of the "lighting fluid," as we named it. This was M. L. Haitinger, whose work on this subject must not be forgotten, and to whom I owe most hearty thanks.

He soon made the interesting observation that the compounds of cerium, which were present in small quantities in the material, were of great importance with regard to the emissive power, and that under certains conditions it was very advantageous to increase the proportion of cerium. He submitted all the mixtures to very exact analysis, and could thus make sure of a good preparation. Eventually I returned to this work and made some hundreds of experiments, without great success, to increase the luminosity of the mixtures. The industry of incandescent gas-lighting was in difficulties, and a continuous sort of stagnation set in; those who had doubted in the beginning again raised their voices, and a very painful time passed for me. Capitalists, mistaken in their hopes, became impatient, and instead of giving me time and leaving me to work quietly, threatened me with legal proceedings. The factory constructed some years before at Atzgersdorf was closed down, and its staff of chemists departed to the four quarters of the globe; finally, I took possession of it and remained there alone.

"Necessity is the mother of invention." It was necessary to achieve something. I started fresh experiments on all possible bodies, but in vain; incandescence by gas did not appear to be susceptible of improvement.

At the commencement of my experiments I had, as I have said, directed my attention to oxide of thorium, and I had observed that it excited the emissive power of other earths. The light given by mixtures having a thorium base was intense, but it did not last. It decreased after about fifty or sixty hours, and was no better then than that from mantles not containing thorium. This astonishing fact made me think that thorium had not yet been examined with sufficient care from the scientific point of view; that is to say, that it contained some substance hitherto unknown to chemists. There chanced to be in the stores at the Atzgersdorf factory a certain quantity of raw, impure thorina. I set myself to work examining this substance, which was then very rare and expensive. I soon found several chemical methods for treating this compound in a much more simple fashion than it had been done before; among others those methods of crystallisation which enabled me rapidly to prepare the pure salts of thorium. A curious fact was then observed, the purer the thorium the less intense was the light given by the mantle made from it. I followed up these experiments, and finally obtained a mantle which only gave a light of two candle power, and I convinced myself by a careful analysis that the thorium which composed it was purer than any of my other preparations. From this I concluded, perhaps a little hastily, that thorium was not an element, but that it could be decomposed. The examination of the mother-liquors showed that they contained the real luminous substance. The decomposition thus seemed to be apparent, but in the mother-liquors from the purest portions I could not find any foreign body. Subsequent experiments, however, enabled me to prove the existence of this luminous compound.

This body, closely allied from many points of view with thorium, and of which the salts were entangled by the salts of thorium, although they were not isomorphous—this body was cerium. The synthesis of the mother-liquors was a simple task; I gradually mixed a solution of cerium with a solution of pure thorium, and I obtained the astonishing light that everyone now knows. The emissive power of the new mixture was about three times that of the old; its life was also much longer, and it opened a new era for incandescent lighting by gas. I made this experiment with the greatest care; I measured the resistance to the action of the air and of the flame; and the decrease in the illuminating power, and at the beginning of the year 1890 I again brought incandescent gas-lighting before the public.

As a general rule the mantles have been kept to this last composition; that is to say, 99 per cent of oxide of thorium and 1 per cent of oxide of cerium.

The value of my patents had then fallen to a minimum, and it was high time that these new results were achieved if I did not want to lose the last few faithful friends who remained to me. Among these I may mention the firm of Julius Pintsch, of Berlin, who were also the first to replace the woven by the knitted mantles, an important improvement I had forgotten to mention.

From the development of incandescent gas-lighting, as I have just described it, it can be seen that my work was not based upon that of others.

When incandescent gas-lighting had arrived at this point the era of lawsuits commenced. Numerous persons, after having rummaged old volumes to find something analogous, came and told me a number of things and names I had never heard mentioned before. I maintain that incandescent gas-lighting was born at Vienna, and that neither I nor my collaborators have ever used the inventions of other people, and I think I have given the best proof of this by what I have said above.

As for this singular property which is possessed by this mixture of 99 per cent of oxide of thorium and 1 per cent of oxide of cerium, it can be seen, it can be observed, but it cannot be explained. This powerful emissive property cannot be foreseen from any property of thorium; this latter hardly possesses it. We can give no exact scientific reason for this excitation of emissive power by cerium, and I expect we shall have to wait a long time before it is explained.

I myself can give no explanation, but I can point out certain analogous facts. These are only ideas, and are not absolutely based on experiments, but still I should like to refer to them briefly.

A body having this emissive power, that is to say, susceptible of emitting an intense light when raised to incandescence in a flame, is composed of infusible oxides in molecular mixture. The principal constituent of this mixture should remain intact in the flame, while the other may be easily reduced and oxidised.

The ponderable relationship between the elements in the mixture should depend on the pressure at which we operate; at the atmospheric pressure this relation is 1 of cerium to 99 of thorium; it might be much more at a pressure of 100 atmospheres.

The gas of the flame successively and very rapidly oxidises and reduces the emissive substance at the points where it is situated in the mantle. If the constituents of the mixture

could form a compound when one of them is in a state of higher or lower oxidation, the compound would be quickly destroyed at the moment when the body concerned passes from the usual to the other degree of oxidation. The earths are in a state of extremely fine division, and are surrounded by a mantle of flame both oxidising and reducing. If reduction takes place there is also decomposition, and if oxidation there is re-combination of these elements; these reactions may go on several million times a second, and molecular shocks are produced which give rise to luminous oscillations of the ether, and the body becomes incandescent.

We may make use of all the substances formed according to this general principle, and for a low consumption of gas they give an intense light. When we examine the whole series of these bodies, of which M. Haitinger has found some, we find another peculiarity which the oxides in the form of salts give to emissive mixtures. These bodies are not chemically but physically analogous; in a certain sense they are isomorphous; that is to say, they crystallise together and form special double salts; the double salts of cerium and thorium are an example. Among the bodies having this emissive power when calcined in molecular mixture we may mention alumina and oxide of chromium. These bodies behave like the oxides of thorium and cerium, only their emissive power is very much less. Oxide of chromium plays the part of the easily oxidisable and reducible oxide, and the alumina that of the infusible basis.

Oxide of chromium and alumina, chromium and aluminium, are not very closely allied from the chemical point of view, still they combine, and each form a series of isomorphous double salts, the alums. Another example is the excitation of this emissive power in lime and strontia by uranium, another discovery of M. Haitinger's.

Small quantities of uranium have the same effect on oxide of thorium. With lime the emissive power of this mixture is very considerable, and for some time we thought of applying it commercially. There again we see the same relations appear. The lime is the infusible basis and the oxide of uranium is the easily reduced and oxidised body. Although these two elements are not closely allied chemically they form certain compounds, of which uranocalcite, which is found in nature, is an example. Their oxides, like their salts, can combine and separate according to the degree of oxidation of the oxide in question. I do not know whether my hypothesis will be acceptable. I have given it because many facts are in its favour, and I know of no observations which are opposed to it. Possibly, again, this explanation might help the development of incandescent gas-lighting.—*Journ. fur Gasbelucht. und Wasservers.*, 1901, p. 661.

The Solubility of Salts. The Sodium Salts of some Bibasic Acids analogous to Sulphuric Acid.—R. Funk.—This paper deals with the comparative solubilities of the neutral sulphate, chromate, molybdate, and tungstate of sodium in their different hydrated forms. All these salts will crystallise in water with 10 H_2O, or anhydrously; the molybdate and tungstate further crystallise with 2 H_2O. The curves of solubility of these salts show a remarkable parallelism. The following are a few of the results obtained at 18°:—

	Density of the saturated solutions.	Molecules of the salt per 100 mols. of water.
$SO_4Na_2+10H_2O$	1·140	2·1
$SeO_4Na_2+10H_2O$	1·315	3·9
$WO_4Na_2+2H_2O$	1·573	4·4
$MoO_4Na_2+2H_2O$	1·437	5·7
$CrO_4Na_2+10H_2O$	1·432	7·4

—*Berichte*, vol. xxxiii., p. 3696.

CONTRIBUTIONS TO THE CHEMISTRY OF THE RARE EARTHS OF THE YTTRIUM GROUP.*

I.

By L. M. DENNIS and BENTON DALES.

Historical.

THE first of the rare earths was discovered by Gadolin (*Sv. Vet. Akad. Handl.*, 1794, p. 137; *Crell's Ann.*, 1796, I., 313), in 1794, in a heavy black mineral which had been found at Ytterby some six years before by Arrhenius. Three years later the earth was named yttria by Ekeberg (*Crell's Ann.*, 1799, ii., 63), and the mineral "yttriastone." The latter is the gadolinite of the present day mineralogists. Ekeberg (*Sv. Vet. Akad. Handl.*, 1802, p. 68; *Ann. de Chim.*, xliii., 278) found yttria in a new mineral in which he had discovered tantalum, and which he called yttrotantalite. The earth was further studied by Berzelius (*Schw. J.*, xvi., 404), and by Berlin (*Sv. Vet. Akad. Handl.*, 1835, pp. 209, 212; *Liebig's Ann. Chem*, xxviii., 222), both of whom considered it homogeneous.

Scheerer (*Pogg. Ann.*, lvi., 482) observed that when yttria was heated strongly in an open vessel it took on a yellow colour, which it lost on heating in the presence of reducing gases, and that it remained colourless if cooled quickly, whereas it became coloured again if heated in contact with air. He thought this phenomenon due to the presence in the yttria of some other earth, perhaps new, perhaps lanthana, capable of forming easily a peroxide. His work was soon followed by the researches of Mosander (*Phil. Mag.*, xxiii., 251; *Liebig's Ann. Chem.*, xlviii., 219), who separated the old yttria into three new earths. He kept the name yttria for the most basic of these; the middle one he called terbia, and the least basic one erbia. His yttria was white, and gave colourless salts; terbia he thought to be white also, but it gave rose-coloured salts, while erbia was orange-yellow and gave colourless salts. He used the methods of fractional precipitation of the oxalates by oxalic acid from acid solution, and of the hydroxides by dilute ammonia, in both of which his erbia was thrown out first, then terbia, and lastly yttria. His results were confirmed by Berzelius ("Lehrbuch," 2nd French Edition, ii., 163), by Svanberg, and by Scheerer.

The earths of old yttria were re-examined in 1860 by Berlin (*Scand. Naturf.* 8 *Möde Kjöbenhavn*, 1860, p. 448). He was the first to use the classic method of fractional decomposition of the nitrates by heat. He obtained only two of Mosander's earths, the white yttria and the one giving rose-coloured salts, terbia, which he and all subsequent investigators have called erbia. Mosander's yellow erbia, the present-day terbia, he could not obtain.

Popp (*Liebig's Ann. Chem.*, cxxxi., 179) in 1864, and Delafontaine (*Arch. des Sci. Phys. et Nat.*, [2], xxi., 97; xxii., 30; xxv., 105; *Liebig's Ann. Chem.*, cxxxiv., 99; cxxxv., 188) in that year and the two following ones, working at this same problem, arrived at totally different conclusions. Popp, using both of Mosander's methods, could separate neither erbia nor terbia. He thought erbia a mixture of the cerite oxides, and terbia a mixture of erbia with yttria. Delafontaine maintained that these two earths existed. Mosander's erbia he obtained by fractional precipitation with primary potassium oxalate, the small amount of yttria and terbia being removed by treatment with potassium sulphate solution. To separate the terbia and yttria remaining, he fractioned again the least coloured oxalates, then dissolved out the yttria by repeated treatment with dilute acid.

Then Bahr and Bunsen (*Liebig's Ann. Chem.*, cxxxvii., 1) in 1866, using a modification of Berlin's method of fusion of the nitrates, corroborated Berlin's results. They obtained only the true or white yttria and the rose-coloured erbia of Berlin (Mosander's terbia). Cleve and Höglund (*Bull. Soc. Chim.*, [2], xviii., 193, 289) in 1872, using the same method, obtained the same result.

* *Journal of the American Chemical Society*, vol. xxiv., No. 5.

Until Mendeleeff's announcement of the periodicity of the elements (*Liebig's Ann. Chem.*, Suppl., viii., 133, esp. p. 184 ff.), yttria, ceria, and the other rare earths had been generally assumed to have the formula RO. He, however, showed that their properties placed them in the third group of his arrangement, where their maximum valence would be three (oxide, R_2O_3). Lanthana he considered to be RO_2. In order to put them into this group, it was necessary to increase their accepted atomic weights by one-half. Cleve (*Bull. Soc. Chim.*, [2], xxi., 344) in 1874, published new researches upon the salts of yttria and erbia, which tended to show the correctness of Mendeleeff's view of the triatomicity of these earths. Nilson (*Ber. d. Chem. Ges.*, viii., 655; ix., 1056, 1142), working upon the selenites in 1875, and upon the chlor-platinum compounds in 1876, further strengthened the idea that the rare earths, lanthana included, were sesquioxides.

A great increase in activity in rare earth research occurred in 1878, when the mineral samarskite, found in large quantities in North America, became the source of the material.

Smith (*Proc. Acad. Nat. Sci. Phil.*, xxix., 194; *Comptes Rendus*, lxxxvii., 146, 148) in 1877 isolated an earth which he considered to be new, or perhaps the third earth of Mosander. He thought it new because of the insolubility of its double potassium sulphate in saturated potassium sulphate solution. Marignac (*Arch. des Sci. Phys. et Nat.* [2], lxiii., 172; *Comptes Rendus*, lxxxvii., 281) showed this property to be only a relative one, and he deemed Smith's mosandria to be identical with terbia. Neither could he agree to Smith's other claim that mosandria was identical with Soret's X. Delafontaine (*Comptes Rendus*, lxxxvii., 600) considered mosandria to be terbia. Considerably later (1886), Lecoq de Boisbaudran (*Comptes Rendus*, cii., 647), having obtained a sample of Smith's impure mosandria, found that it contained, besides didymia and samaria, gadolinia and terbia.

Delafontaine (*Arch. des Sci. Phys. et Nat.*, [2], lxi., 273; *Ann. Chim. Phys.*, [5], xiv., 238) and Marignac (*Arch. des Sci. Phys. et Nat.*, [2], lxi., 283; *Ann. Chim. Phys.*, [5], xiv., 247) settled the question as to the existence of terbia by preparing it. Delafontaine treated his yttria earth mixture in solution with a saturated solution of sodium sulphate containing crystals of the salt, then fractioned the precipitated portion with oxalic acid from strong nitric acid solution, and finally treated the first of these precipitates with formic acid and concentrated. Terbia crystallised out as formate. There was also a yellow earth in the portion soluble in the sodium sulphate, but it was not so deeply coloured as terbia, and gave besides a base of lower atomic weight. He thought it a new element. Marignac obtained terbia as well as yttria and erbia by fusion of the nitrates. By making several hundred fractions and by properly combining them he obtained finally pure yttria at one end and erbia at the other. The intermediate products possessed a yellow colour. These he combined and fractioned by oxalic acid in acid solution. Erbia and didymia he found to go with terbia. Didymia he removed by the potassium sulphate treatment, but he could not free his product from erbia. Hofmann and Krüss (*Zeit. Anorg. Chem.*, iv., 27) in 1893 studied terbia, and by fractionation with aniline hydrochloride succeeded in breaking it up into two probable earths with metal atomic weights of 148 to 150 and 160, calculated as R^{III}. In 1895 Lecoq de Boisbaudran (*Comptes Rendus*, cxxi., 709) called attention to an absorption band in his terbia material which could not belong to any known element. He called the one which gives it Z_β. Its band was at λ 487·7.

The new earth which Delafontaine suspected in the saturated sodium sulphate solution mentioned above, was announced by him (*Comptes Rendus*, lxxxvii., 559) as philippia. It was characterised by a magnificent absorption band in the violet at λ 450. Soret (*Comptes Rendus*, lxxxix., 521) stated that philippia was identical with the earth X, which Cleve denied (*Comptes Rendus*, lxxxix., 708). Delafontaine (*Comptes Rendus*, xc., 221) maintained that philippia, Soret's X, and Cleve's holmia were identical, which Cleve again denied. Delafontaine (*Arch. des Sci. Phys. et Nat.*, [3], iii., 246) then admitted that the absorption spectrum ascribed by him to philippia belonged to Soret's X, but maintained still the individuality of the former. Roscoe (*Ber. d. Chem. Ges.*, xv., 1274) in 1882, by fractional precipitation of the double potassium sulphates with potassium sulphate, and by fractionation of the first portions of these by the varying solubilities of the formates, could obtain no product whose metal had a constant atomic weight of 121 to 123, and he concluded that philippia was a mixture of terbia and yttria. Crookes (*Phil. Trans. Roy. Soc.*, clxxiv., 910) agreed with Roscoe as to the non-existence of philippia. In 1897, Delafontaine (Chem. News, lxxv., 229) gave the methods by which philippia might be prepared; they were the standard ones of fractional precipitation of the nitrate solution of the rare earths with ammonia, or with primary potassium oxalate, or fractional decomposition of the nitrates by heat. Urbain (*Ann. Chim. Phys.*, [7], xix., 192), in 1900, denied the existence of philippia.

In this year of 1878, while studying the impure didymia from samarskite, Delafontaine (*Comptes Rendus*, lxxxvii., 632) discovered a new oxide which he called decipia. Its metal had a high atomic weight (R^{III}=159), and it was characterised by the absorption bands λ 416 and λ 478. Lecoq de Boisbaudran (*Comptes Rendus*, lxxxviii., 322; lxxxix., 212) found, in a spectroscopic examination of a mixture of earths rich in didymia from samarskite, some new rays. In the spark spectrum there were four rays; in the absorption spectrum, two strong bands and three faint ones. The strong bands were in the blue, and had their centres at λ 480 and λ 463·5. One of the other bands had a wave-length of λ 416, which had been ascribed by Delafontaine to one of the bands of decipia. Lecoq de Boisbaudran finally separated the earth giving these bands from didymia, and he called it samaria. Then Marignac (*Arch. des Sci. Phys. et Nat.*, [3], iii., 413), by fusion of the nitrates and fractionation of the double potassium sulphates, isolated two earths which he called Y_α and Y_β. The double potassium sulphate of Y_α was comparatively soluble in potassium sulphate solution; solutions of the earth gave no absorption spectrum, and the equivalent weight of the metal was about 120·5 (RO). Y_β, on the other hand, gave a double potassium sulphate less soluble in potassium sulphate solution; solutions of the earth gave an absorption spectrum corresponding to that of decipia, or better to that of samaria, and the metal had an equivalent weight of 115·6. Y_α has since been called gadolinium (*Comptes Rendus*, cii., 902). Delafontaine (*Comptes Rendus*, xciii., 63) again in 1881 showed that, by treating those earth sodium double sulphates most insoluble in a saturated solution of Glauber's salt with cold water, his decipia of two years before could be decomposed into two oxides, one with a metal equivalent of about 130 (RO) giving no absorption spectrum, the other with a base of much lower equivalent (not over 117) giving the absorption spectrum ascribed to the original decipia. He expressed the opinion that this second oxide and samaria were identical, as well as Marignac's Y_β. He also thought that the earth Y_α was a mixture of decipia and terbia, but Marignac pointed out that this could not be, because the oxide of Y_α was white, whereas that of terbium was coloured. The oxide of Y_α and decipia were perhaps identical.

Cleve (*Comptes Rendus*, xcvii., 94; *Bull. Soc. Chim.*, [2], xliii., 162; Chem. News, liii., 30) has made an exhaustive study of samaria and its compounds. He isolated it by combining the methods of fractional precipitation with potassium sulphate solution, and with dilute ammonia. The atomic weight of samarium he found to be 150·02 (R^{III}). Its spark spectrum has been described by Thalén (*Sv. Vet. Akad. Handl.*, 1883, No. 7, p. 3; *Journ. de Phys.* [2], ii., 446; *Ber. d. Chem. Ges.*, xvi., 2760), and by Lecoq de Boisbaudran (*Comptes Rendus*, cxiv., 575;

cxvi., 611, 674; cxvii., 199). The latter also studied its fluorescence spectrum. The body giving the spark spectrum he called Z_α, the one giving the fluorescence Z_ζ. Demarçay (*Comptes Rendus*, cii., 1551) found that samarium could be split into at least two simpler constituents. He used three methods, Welsbach's, fractional precipitation with ammonia, and another not given. He kept the name samarium for the element giving λ 407 and λ 400, and called the other provisionally S. Krüss and Nilson (*Ber. d. Chem. Ges.*, xx., 2134), in an article on the rare earths giving absorption spectra, also observed the fact that solutions containing samarium did not always give the same absorption spectrum. They obtained some solutions showing only λ 417, and the body to which this band was due they called Sm_α. Crookes (*Proc. Roy. Soc.*, xl., 502) in 1886, following the fractionation of samarium by examination of the phosphorescence spectra, concluded that the samarium of gadolinite enclosed three components, while the samarium from samarskite contained a fourth. He also observed an anomalous ray in the phosphorescence spectrum of samarium (*Comptes Rendus*, cii., 1464). Its wavelength was 609, and the body to which it was due he called S_α. In 1891 Bettendorff (*Liebig's Ann. Chem.*, cclxiii., 164) obtained pure samaria by fractionation with ammonia, after the removal of ceria by fusion of the nitrates with potassium nitrate, the yttria group by treatment with potassium sulphate, and lanthana by decomposition of the nitrates by heat. He described the absorption spectrum, and denied that it had a phosphorescence one. He thought that samarium was a chemical individual. In 1893 Demarçay (*Comptes Rendus*, cxvii., 163) decided that, as far as the absorption spectrum showed, there was no reason to suspect the complexity of samarium. He examined four different fractions of Lecoq de Boisbaudran's samarium material. In 1900 Demarçay (*Comptes Rendus*, cxxx., 1185) separated samaria from neodidymia by fractional crystallisation of the magnesia double nitrates from nitric acid.

Crookes (Chem. News, liv., 39, 115) questioned the individuality of gadolinium, stating that its phosphorescence spectrum was practically identical with that of a mixture of 61 parts of yttria and 39 of samaria. Lecoq de Boisbaudran (*Comptes Rendus*, cviii., 165; cxi., 393) did not agree with him. Lecoq found, however, that Marignac's gadolinia contained about 10 per cent of impurities, and he succeeded in removing all but about 0·02 or 0·03 per cent of these. Crookes (*Comptes Rendus*, cii., 646) described its phosphorescence spectrum, and Lecoq (*Comptes Rendus*, cxi., 472) its spark spectrum. Bettendorf (*Liebig's Ann. Chim.*, cclxx., 376) separated gadolinia from samaria and terbia by fractionation with ammonia. He could get no spark spectrum, nor could Thalén. In 1896 Demarçay (*Comptes Rendus*, cxxii., 728) prepared gadolinia by fractional crystallisation of the earths rich in samaria from fuming nitric acid (sp. gr., 1·45), and gave the rays of its spark spectrum. Gadolinia was precipitated first, and samaria last. Between the two he discovered a new earth which he called Σ, characterised by its colourless salts with no absorption. The earth was colourless, which distinguished it from terbia; its spark spectrum was different from those of lanthana, ceria, gadolinia, ytterbia, terbia. It differed from gadolinia and samaria only in its spark spectrum. In 1899 Benedicks (*Zeit. Anorg. Chem.*, xxii., 393), using Marignac's method partly, and partly by fusion of the nitrates, then their fractionation from concentrated nitric acid, and lastly their precipitation by ammonia, prepared gadolinia and studied it. He could not obtain Demarçay's Σ. His gadolinia gave a spark spectrum, and the atomic weight of the metal was 156 (R^{III}). Demarçay (*Comptes Rendus*, cxxx., 1019), in 1900, was unable to isolate Σ, even after 800 fractions with the magnesia double nitrates; but since then he has isolated it sufficiently pure for characterisation (*Comptes Rendus*, cxxxii., 1484). He has named it europium.

Marignac (*Arch. des Sci. Phys. et Nat.*, [2], lxiv., 97; *Comptes Rendus*, lxxxvii., 578), in trying to isolate Delafontaine's philippia by partial decomposition of the nitrates by heat, discovered a new earth which he called ytterbia. It was colourless, and its metal had a high atomic weight (R^{III} = 172·5); further, it had no absorption spectrum. The method he used was a modification of Bahr and Bunsen's. He heated the nitrates till a portion was insoluble in hot water, whereas they heated only until the mass, which was entirely soluble in hot water, gave a crystalline precipitate of basic nitrates on cooling. In 1879 and 1880, Nilson (*Ber. d. Chem. Ges.*, xii., 550; xiii., 1430), using Marignac's method, isolated more ytterbia, and studied it. He found the atomic weight of the metal ytterbium to be 173. Pure ytterbia was obtained only after 400 or 500 ignitions. While working with this material, he obtained a white oxide whose metal equivalent was lower than that of ytterbia. This caused him to suspect the presence of another oxide, the metal of which had a lower atomic weight. By following the same method of fractionation, he succeeded in isolating this earth (*Ber. d. Chem. Ges.*, xii., 554; xiii., 1439). Its base had an atomic weight of 44, and proved to be identical with Mendeleeff's ekaboron, both in its properties and its atomic weight. He called his earth scandia. Cleve (*Bull. Soc. Chim.*, [2], xxxi., 486) also prepared scandia. His atomic weight of scandium was a unit higher than Nilson's, probably because of ytterbium in the material. The spark spectrum of ytterbia has been studied by Thalén (Chem. News, xlvii., 217; *Comptes Rendus*, xci., 326), and by Lecoq de Boisbaudran (*Comptes Rendus*, lxxxviii., 1342); and that of scandia by Thalén (*Comptes Rendus*, lxxxviii., 646; xci., 45; Chem. News, xlvii., 217).

(To be continued).

ANALYSIS OF THE VOLCANIC DUST FROM THE RECENT ERUPTION IN THE WEST INDIES.

We are indebted to the kindness of Mr. B. E. R. Newlands for the following analysis of the volcanic dust which fell over the Barbados from La Soufrière, on May 8th, 1902:—

SiO_2	51·60
Al_2O_3	21·12
Fe_2O_3	9·28
CaO	9·07
MgO	3·96
Na_2O	0·59
K_2O	0·81
SO_3..	Undetermined
P_2O_5	do.
	96·43

We hope next week to give the analyses of two similar dusts, one from Mont Pelée, which fell on the deck of the *Roddam*, and the other collected at Grenada.

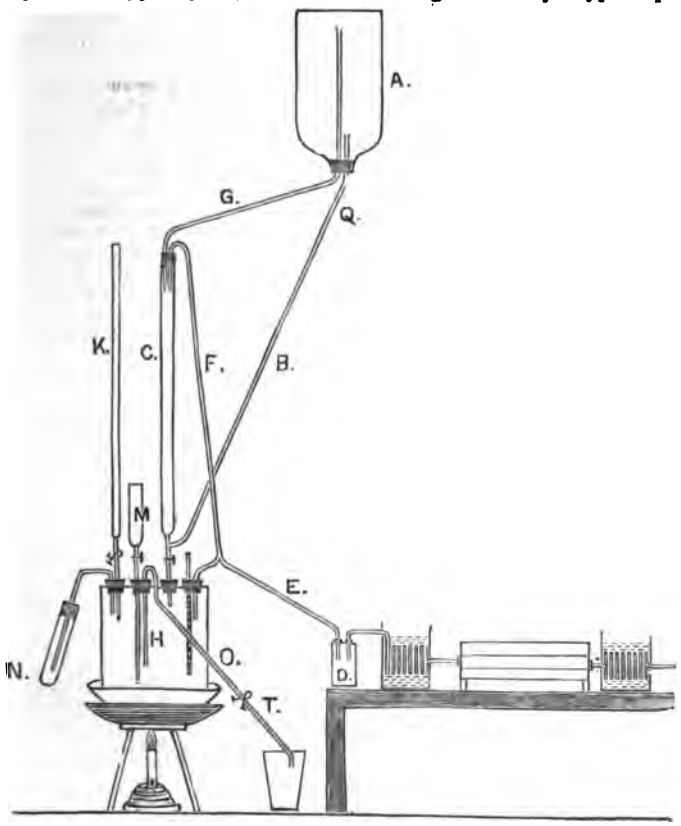
Society of Public Analysts.—Testing for Arsenic.—The Secretary of the Royal Commission on Arsenical Poisoning having expressed the desire of the Commission to know to what extent the method of testing for and estimating minute traces of arsenic prescribed by the Joint Committee of the Society of Chemical Industry and of the Society of Public Analysts has been adopted, and with what results, the Secretaries of the above Society have issued a slip containing four questions which they request may be replied to as concisely and briefly as possible and returned to them. The questions are:—1. Have you used the method; and if so, to what extent? 2. Have you found it advantageous, and do you consider it preferable to other methods? 3. Have you met with any, and if so what, difficulties? 4. Have you any suggestions to offer with regard to the better working of the method?

THE ESTIMATION OF DISSOLVED OXYGEN IN WATER.

By WILLIAM NAYLOR, F.C.S., A.M.I.C.E.

The estimation of dissolved oxygen in river water and sewage works effluents has now become quite common, and the following method, devised by B. W. Gerland, as practised by the author, may be of interest. Though not applicable for river-side estimations, nor sufficiently "handy" for single estimations, it has been found very serviceable for the work of a station where a great number of samples, say 40 or 50, have to be examined in one day.

Its principle, as described by Dr. Gerland (*Journ. Soc. Chem. Ind.*, xv., p. 15; xviii., p. 340) is the reduction of indigo by sodium hyposulphite, and this is now brought into daily quantitative use owing to the accessibility of pure indigo, and the tedious process of standardising against aërated water avoided.



The pure indigo, obtainable from the Badische Aniline and Soda Works, Ludwigshafen, after washing with water and hydrochloric acid and alcohol, and treating with a solution of persulphate of ammonia in acid solution, will be found, after reduction, to require the theoretical amount of oxygen for oxidation.

In examining a sample of water, the four-necked Woulff's bottle, H, is first charged with a small quantity, about 50 to 100 c.c., of tap water by means of the thistle funnel, M, and a stream of coal-gas which has been passed through a heated copper tube, filled with copper turnings, is sent also into the bottle via the tube E and out by water seal, N.

A branch, F, from the tube E conveys some of this de-oxidised coal-gas to top of hypo reservoir, A, and burette C. Into the water in the bottle are now run one or two c.c. of indigo solution, and these are reduced exactly by hypo from the burette, C, which burette is charged from time to time by means of the tube B, having clip at Q. With the indigo reduced in the Woulff's bottle, the liquid will remain a pale straw-colour if the atmosphere is oxygen free, and as soon as this is the case the hypo is standardised against the indigo solution.

After standardising, the contents of the bottle are at neutral tint, and about 100 c.c. of the sample under examination are passed in by means of the thistle funnel, M, provided with tap, and the quantity of hypo required noted, as a guide. It is important that the funnel dip right into the liquid to prevent any yield of oxygen to the atmosphere of coal-gas.

With the contents at neutral tint after discovering the hypo required for 100 c.c. of sample, a little under two and a-half times this amount of hypo is now run into the bottle, and then 250 c.c. of the sample. A faint blue colour is produced, and this is removed by the addition of a drop or two of hypo and the total for the 250 c.c. of sample entered.

Any number of samples can be done in succession, the bottle being drawn off as necessary by means of the tube O and clip T.

The following are two note-book entries, the first of which was found to be in agreement with standard aërated water as per table of Roscoe and Lunt, and also with a eudiometric estimation after boiling from a completely filled flask and running in warm mercury:—

10 c.c. indigo solution (neutralised with sodium bicarb. before use) required 24·5 c.c. hypo.

Indigo sol. contained 8·321 grms. indigotine per litre.

1 c.c. indigo sol. contained available oxygen 0·0005.

24·5 c.c. hypo = 0·005 oxygen.

250 c.c. sample of aërated water required 12·5 c.c. hypo = 0·0025 grm. oxygen.

1000 c.c. = 0·01 grm. oxygen.

$$\frac{0{\cdot}01 \times 1000}{1{\cdot}4336} = 7{\cdot}0 \text{ c.c. oxygen per litre.}$$

Bacterial Filter Effluent, after shaking to saturation.

10 c.c. indigo sol. required 37·0 c.c. hypo.

250 c.c. sample required 10·0 c.c. hypo = 0·00135 grm. oxygen.

1000 c.c. sample = 0·0054 grm. oxygen.

$$\frac{0{\cdot}0054 \times 1000}{1{\cdot}4336} = 3{\cdot}77 \text{ c.c. oxygen per litre.}$$

The method is not nearly so complicated or cumbersome as it appears at first glance, and the apparatus once set up will give very little trouble. In an investigation just completed, on "The Movement of the Polluted Zone in the Ribble Estuary," it was found that a hundred samples could be examined in one day, without any necessity for washing out the Woulff's bottle.

Ribble Joint Committee's Laboratory,
Preston, May 20, 1902.

Estimation of Starch in the Grains of Cereals.—M. Lindet.—This method is based on the attack of the grains by means of hydrochloric pepsine, which dissolves the gluten, on the kneading of the mass in a silk bag, and on the collection of the starch. The author has modified the latter part of the estimation, and replaced it by decantation and saccharification, after which the glucose and dextrine formed are weighed.—*Bull. Soc. Chim. de Paris*, xxv., No. 24.

PROCEEDINGS OF SOCIETIES

CHEMICAL SOCIETY.

Ordinary Meeting, Thursday, May 15th, 1902.

Dr. W. H. PERKIN, F.R.S., Vice-President, in the Chair.

CERTIFICATES were read for the first time in favour of Messrs. James Handby Ball, St. Stephen's Villa, Limerick; Bryce Chudleigh Burt, University Settlement, Bermondsey, S.E.; Arthur Crozier Claudet, 27, Daleham Gardens, Hampstead, N.W.; Edgar William Foll, 40, Sutton Street, Lambeth, S.E.; John Longsdon Garle, 136, Holland Road, Kensington, W.; Geo. H. Leader, Sexey's School, Blackford, Somerset; James Butler Moody, 111, Manchester Road, Burnley, Lancs.; Percy Philip Phillips, Haslemere, Morris Avenue, Manor Park, E.; William E. F. Powney, 67, Barretts Grove, Stoke Newington, N.; William C. S. Stanger, 72, Belle Vue Road, Ipswich.

Of the following papers, those marked * were read:—

*74. "*The Variation with Temperature of the Surface-tensions and Densities of Liquid Oxygen, Nitrogen, Argon, and Carbon Monoxide.*" By E. C. C. BALY and F. G. DONNAN.

The measurements were made by the method of elevation in capillary tubes. The surface tensions, molecular surface-energies, and densities of liquid oxygen, nitrogen, argon, and carbon monoxide have been determined between the temperatures of 70° and 90° (absolute). In the case of argon, owing to the proximity of its freezing- and boiling-points, measurements could only be made between 84° and 89°.

The relation between molecular surface energy and temperature is found to be linear, thus agreeing with the results obtained by Eötvös and by Ramsay and Shields for non-associated liquids.

The values found for $\frac{d}{dt}\gamma(Mv)^{\frac{2}{3}}$, the temperature coefficient of the molecular-surface energy, are as follows:—

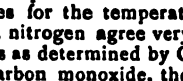
Oxygen	1·917
Nitrogen	2·002
Argon	2·020
Carbon monoxide . ..	1·996

The average value obtained by Ramsay and Shields for non-associating liquids was 2·212.

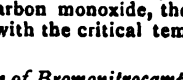
Continuations of the straight lines connecting molecular surface energy and temperature so as to cut the temperature axis give values for the temperature which in the case of oxygen and nitrogen agree very closely with the critical temperatures as determined by Olszewski. In the case of argon and carbon monoxide, the temperatures so found do not agree with the critical temperatures as previously determined.

*75. "*Comparison of Bromonitrocamphane with Bromonitrocamphor.*" By M. O. FORSTER.

The close relationship between 1 : 1-bromonitrocamphane,—



and *aa*-bromonitrocamphor,—



led to the expectation that the series of changes by which the former substance is converted into a hydroxylic isomeride of camphor might be applied to bromonitrocamphor, and thus give rise to a hydroxylic isomeride of camphorquinone. Bromonitrocamphor was therefore heated with alcoholic silver nitrate until silver bromide was no longer precipitated. This treatment, which transforms bromonitrocamphane into nitrocamphene, $C_{10}H_{15}\cdot NO_2$, proceeds very slowly in the case of the camphor derivative, the solution becoming yellow. Instead of the expected nitro-compound, $C_{10}H_{13}O\cdot NO_2$, the only crystalline product obtained was camphorquinone. The yield of quinone by the new method amounted to 25 per cent; the process therefore compares favourably with that of Claisen, but is more tedious.

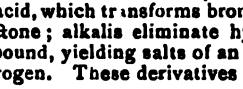
The comparison of bromonitrocamphane with bromonitrocamphor has been extended to the behaviour of these substances towards phenylhydrazine. The uncontrolled action of the base is exceedingly vigorous and yields no definite product; under regulated conditions, however, bromonitrocamphane is reduced to nitrocamphane, which may be thus obtained in a purer form than has been possible hitherto. Nitrocamphane prepared by this method melts at 157° instead of 147—148°, and has $[\alpha]_D = +27{\cdot}0°$ in benzene, and +7·4° in absolute alcohol, instead of 20·4° and 4·6° respectively.

In the case of bromonitrocamphor, phenylhydrazine gives rise to nitrocamphor in a form which requires to be crystallised only once from petroleum to produce the substance in the colourless state, melting at 103° and giving $[\alpha]_D = -123{\cdot}8°$ in benzene; as the yield amounts to 84 per cent of the theoretical, this method appears to be a distinct improvement on the existing process.

Bromine has no action on bromonitrocamphor under conditions which lead, in the case of bromonitrocamphane, to elimination of the nitro-group and production of a tribromo-derivative. Bromonitrocamphor is also indifferent towards nitric acid, which transforms bromonitrocamphane into a new lactone; alkalis eliminate hydrogen bromide from this compound, yielding salts of an unsaturated acid containing nitrogen. These derivatives are being investigated.

*76. "*aa-Benzoylnitrocamphor and aa-Benzoyliodocamphor.*" By M. O. FORSTER and E. A. JENKINSON.

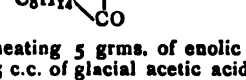
aa-Benzoylnitrocamphor,—



prepared by heating 5 grms. of enolic benzoylcamphor dissolved in 25 c.c. of glacial acetic acid with 1·5 c.c. of fuming nitric acid, is also produced when nitrogen peroxide is passed into a solution of benzoyl camphor in dried chloroform. It requires 40 parts of boiling alcohol to dissolve it and is sparingly soluble in common solvents, crystallising in rectangular plates which melt and evolve gas at 225°. A 2 per cent solution in chloroform has $[\alpha]_D = +245{\cdot}2°$. It is insoluble in aqueous alkalis, is indifferent towards ferric chloride, and does not give Liebermann's reaction.

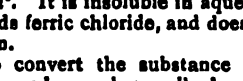
Attempts to convert the substance into *aa*-benzoylaminocamphor and *aa*-benzoylhydroxylaminocamphor have been unsuccessful, but it is readily hydrolysed by alcoholic potash, yielding α-nitrocamphor and benzoic acid.

aa-Nitrobenzoylnitrocamphor,—



produced when benzoylnitrocamphor is dissolved in fuming nitric acid, is more readily soluble than the foregoing substance, and crystallises from alcohol in pale yellow leaflets which melt and evolve gas at 176—177°; a 2 per cent solution in chloroform has $[\alpha]_D = +190{\cdot}6$. Hydrolysis with alcoholic potash resolves the substance into nitrocamphor and *m*-nitrobenzoic acid.

aa-Benzoyliodocamphor,—



is not obtainable by the methods which give rise to the benzoylbromocamphors and benzoylchlorocamphors; it is

prepared by adding a solution of iodine in potassium iodide to enolic benzoylcamphor dissolved in aqueous potash. The compound crystallises from alcohol in pale yellow plates which become reddish brown when exposed to light; it melts at 136°. A 2 per cent solution in chloroform has $[\alpha]_D = +47{\cdot}7°$.

Benzoyliodocamphor is not converted into an isomeride by the action of hydrobromic acid, and hydriodic acid merely reduces it. Solutions in organic liquids rapidly undergo decomposition, with liberation of iodine; ferric chloride soon produces a pale green colour, becoming bluish, and finally purple. Attempts to convert the substance into benzoylcyanocamphor and benzoylhydroxycamphor have been unsuccessful.

*77. "2 : 4-*Dibromo-5-nitro-* and 2 : 4-*Dibromo-*3 : 5-*dinitro-toluenes and their Behaviour on Reduction.*" By W. A. Davis.

On nitration with fuming nitric acid, 2 : 4-dibromotoluene gives 2 : 4-dibromo 5-nitrotoluene, which is converted by reduction with tin and hydrochloric acid into 4 : 6-dibromo *m*-toluidine, the *acetyl*-derivative of which crystallises from alcohol in small white prisms (m. p. 167°).

If the nitration of 2 : 4-dibromotoluene be not carefully regulated, 2 : 4-*dibromo*-3 : 5-*dinitrotoluene* is obtained. It crystallises from ethyl acetate in yellow prisms (m. p. 127·5°), and differs from all the other bromo- and dibromonitrotoluenes, as it loses the whole of its bromine on reduction with tin and hydrochloric acid, giving *s*-toluenediamine; the *diacetyl*-derivative, $C_6H_3Me(NHAc)_2$, crystallises from alcohol in small hemimorphic prisms (m. p. 236—237°) and is strongly electric. The removal of the bromine atoms from 2 : 4-dibromo-3 : 5-dinitrotoluene during reduction is apparently due to the influence of the two contiguous nitro-groups.

*78. "*Note on the Purification of Hydrochloric Acid from Arsenic.*" By L. T. Thorne, Ph.D., and E. H. Jeffers.

In the estimation of arsenic by the Marsh-Berzelius test, the use of hydrochloric acid is admittedly preferable to that of sulphuric acid. It is, however, practically impossible to purchase hydrochloric acid really free from arsenic, and it is therefore necessary to remove the last traces of arsenic from it in the laboratory.

The method recommended in the report of the Joint Committee of the Society of Chemical Industry and of Public Analysts (*Analyst*, 1902, xxvii., 48), namely, that of treatment of the diluted acid with hydrobromic acid or bromine and sulphurous acid and subsequent distillation, is successful in most cases, but occasionally fails; the arsenic then appearing to be present in a very stable or resistant form. Under these latter conditions, satisfactory results are said to be obtained if the hydrobromic and sulphurous acids are added to the *strong* hydrochloric acid, and the excess of hydrochloric acid gas boiled off before distillation commences. If large quantities of hydrochloric acid are wanted, this method is inconvenient and wasteful, owing to the large quantities of hydrochloric acid gas evolved.

The whole of the arsenic may readily be removed by means of a modification of the well-known Reinsch test for arsenic in the following manner:—

The hydrochloric acid to be purified is diluted with water till its specific gravity is about 1·10, then raised to the boiling-point, and a piece of fine copper gauze introduced. The gauze should be of about 100 meshes to the inch, and of as pure copper as possible. When a couple of litres of liquid are being dealt with, the gauze should be about four inches square and coiled very loosely (about 1½ to 2 turns) round a long glass rod flattened at the end, as in this way it can be readily introduced and removed, besides being brought into better contact with the liquid. The whole is then kept just boiling for an hour. By this time the gauze will have become more or less blackened, and should be replaced by a second piece and the digestion continued for another hour. If this piece is also blackened, a third is used, and so on, until the gauze remains perfectly bright after about an hour's digestion. With moderately good acid, two, or at most three, pieces of gauze are sufficient. The gauze is then removed, and the acid at once transferred to a retort and distilled from a fresh piece of gauze. It is advisable, as a precaution, to reject the first 20 per cent of the distillate, though even this is very rarely contaminated if the digestion is carefully carried out, and from 100 to 200 c.c. should be left behind in the retort. The method is simple and requires but little attention, and it is not necessary to use the purest acid; even good muriatic acid, "commercially free from arsenic," may be satisfactorily employed. The acid obtained is the constant boiling-point acid, which is about the most suitable strength for use in the Marsh-Berzelius test.

The gauze must, of course, be gently ignited before being used, and should not be introduced until the acid is just beginning to boil. It is also preferable not to allow the digested liquid to cool in contact with the air before transferring to the retort, as the copper gauze would be much attacked and its efficiency for the present purpose considerably lessened by the consequent oxidation.

79. "*The Radio-activity of Thorium Compounds.* Part II. *The Cause and Nature of Radio-activity.*" By E. Rutherford and F. Soddy.

The authors find that thorium from which ThX has been separated regains its activity with time, whilst the activity of ThX decreases with time. At the end of three weeks, the activity of the former again reached a maximum value, and the activity of the latter almost completely disappeared.

ThX possesses a distinct chemical behaviour which differentiates it from thorium. Ammonia is the only reagent of those tried capable of separating it from the latter. Ammonium carbonate, oxalic acid, and sodium phosphate give precipitates with normal activity, and the residues from the filtrates are inactive.

Using the radio-activity of the residues from the filtrates after precipitation of the thorium by ammonia, as a means of determining the amounts of ThX present, it was found that the amounts produced in varying intervals of time between successive precipitations agree with the requirement that ThX is being continuously produced by thorium compounds at a constant rate.

The rate of production of ThX and the rate of decay of its activity are apparently unaffected by known agencies. Both changes proceed independently of the chemical and physical conditions of the molecule.

The source of the energy required to maintain the radio-activity of thorium over indefinite periods is therefore to be found in a chemical change producing new types of matter.

Many of the results obtained by the authors in their investigation of the thorium emanations are now capable of explanation. Emanating power appears as a property of ThX and not of thorium, and is proportional to the activity of the ThX present. The decay and recovery of emanating power of ThX and thorium are completely analogous to the decay and recovery of radio-activity. These results find their simplest explanation in the view that a secondary change is proceeding in ThX. One of the products is gaseous, and in the radio-active state constitutes the emanation. This change appears more allied to ordinary chemical reaction than the primary, for it is affected by the conditions.

The residual activity of thorium would be explained if the chemical change which produces ThX produces also a second kind of active matter, closely allied to thorium in its properties. The radiations of this residual part are composed entirely of rays non-deviable in the magnetic field, whereas the other two components of thorium radio-activity comprise both deviable and non-deviable radiation. In this respect uranium and thorium are analogous (compare following paper).

The present result, that radio-activity is the consequence of changes—independent of the conditions—in which new types of matter are formed, leads to the conclusion that radio-activity is the manifestation of sub-atomic chemical change.

80. "*The Radio-activity of Uranium.*" By F. Soddy.

During an investigation of the radio-activity of uranium (conjointly with Prof. Rutherford) great differences arose between the results obtained and those of previous workers. These are traced to the methods employed for measuring radio-activity.

UrX prepared by Crookes's original method (*Proc. Roy. Soc.*, 1900, lxvi., 409), although intensely active to the photographic plate, is almost inactive to the electrometer under ordinary circumstances. The uranium from which it is separated is inactive to the sensitive film, but its activity to the electrometer is very nearly normal.

This is due to the dual character of uranium radiation (Rutherford, *Phil. Mag.*, 1899, [5], xlvii., 109). The α- or easily absorbed radiation is without appreciable action on the photographic film, and contributes by far the major portion of the ionisation effect observed by the electrometer. The β- or penetrating radiation, on the other hand, causes the whole of the photographic effect, but being little absorbed by gases, and constituting but a small proportion of the total radiation, its ionisation effect cannot be well observed by the electrometer without special arrangements.

UrX possesses all the β- with none of the α-radiation, the latter being completely retained by the uranium. The β- is wholly deviable in the magnetic field, the α-radiation not at all. This explains the conclusion of Becquerel (*Comptes Rendus*, 1902, cxxxiv., 208), who worked solely with the photographic method, that the whole of the uranium radiation is deviable in the magnetic field.

The discovery of Becquerel that inactive uranium recovers its activity with time (*Comptes Rendus*, 1901, cxxxiii., 977) therefore points to a continuous production of UrX by the uranium. This continuous production can be observed by means of the β-radiation in uranium originally freed from UrX after three days.

The published methods of Crookes (*loc. cit.*) and Becquerel (*Comptes Rendus*, 1900, cxxxi., 137) fail to remove the α-radiation of uranium, or even to diminish its quantity. As in the case of thorium, this constitutes a residual activity comprising only non-deviable radiation, and it has not yet been found possible to remove it by chemical means. Experiment shows that if it is a secondary radiation caused by UrX, it must take over a year to decay to half its value after the exciting cause is removed. It seems therefore more probable that it is caused by a second type of non-uranium matter, produced from the uranium by the same change as the UrX.

Research Fund.

A meeting of the Research Fund Committee will be held in June. Applications for grants, to be made on forms which can be obtained from the Assistant Secretary, must be received on or before June 9th.

NOTICES OF BOOKS.

A Bibliography of the Analytical Chemistry of Manganese, 1785-1900. By Henry P. Talbot and John W. Brown. Smithsonian Miscellaneous Collections, 1313. City of Washington, 1902. 8vo. viii.-124 pp.

This forms one of the series of Indexes to Chemical Literature prepared under the auspices of the Committee of the American Association for the Advancement of Science, and recommended by them to the Smithsonian Institution for publication. The compilers in their preface acknowledge their indebtedness to a similar work by H. Carrington Bolton published twenty-five years ago, and to Brearley's "Bibliography of Steel Works Analysis" in the Chemical News. The periodicals examined number fifty-four, comprising several hundred volumes. The references are arranged chronologically, and are followed by a full subject-index and an author-index, the two occupying 27 pages, double columns. The work will prove of value to analytical chemists, especially to those desirous of becoming acquainted with the results of the labours of their predecessors. Students of the history of analytical methods will also find this invaluable. Its handsome paper and typography does credit to the Smithsonian Institution and to the printers.

Index to the Literature of the Spectroscope (1887-1900, both inclusive). Continuation of the previous Index, by the same Author, published in 1888. By Alfred Tuckerman. Smithsonian Miscellaneous Collections, 1312. City of Washington, 1902. 8vo. iii.-373 pp.

This second volume of Dr. Tuckerman's well-known bibliography brings the literature down to the end of the nineteenth century, after which date the International Committee for Indexing Scientific Literature will continue the work.

The arrangement is satisfactory: first, the reference to literature is placed under the author's name, the latter in alphabetic order; and, secondly, a classified subject-index, in which the references are briefly repeated. Of course, it is often difficult to select topics that should be included or excluded, and we notice a few that seem to be out of place; on the whole, it is better to err by including foreign matter than by excluding the pertinent factors.

The thanks of all physicists and chemists are due to Dr. Tuckerman for continuing this arduous labour of love—for such it has been—and to the Smithsonian Institution for the handsome dress in which it appears.

Indicators and Test-papers; their Source, Preparation, Application, and Tests for Sensitiveness. A *résumé* of the Current Facts regarding the Action and Application of the Indicators and Test-papers which have been proposed from time to time, and are in present use in Chemical Manipulations. With a Tabular Summary of the Application of Indicators. Designed for the Use of Chemists, Pharmacists, and Students, by Alfred I. Cohn. Second Edition, Revised and Enlarged. New York: John Wiley and Sons. London: Chapman and Hall, Lim. 1902. 12mo. Pp. ix.-267.

The first edition of this useful little book was noticed in the Chemical News for November 3, 1899 (vol. lxxx., p. 219). Since then some errors have been corrected, and the entire work has been brought down to date by the addition of an appendix embodying the information on the indicators introduced in the intervening years. Perezol is one of the new indicators (of Mexican origin) perfectly adapted for titrating alkalis, reacting with great delicacy; it is particularly useful in estimating the alkalinity of potable waters, &c.

In each section, Indicators and Test-papers, an alphabetic arrangement is adopted, making it easy to refer to a given topic. There is an uncommonly full index.

H. C. B.

Class List and Index of the Periodical Publications in the Patent Office Library. London: Darling and Son, Ltd., and The Patent Office. 1902. Pp. 191.

The heading "Periodical Publications" includes Journals, Transactions of Societies, Year-books, Reports of Congresses, Departments, &c.

This handbook is divided into three sections—(*a*) An abstract of the classifications adopted; (*b*) a classified catalogue; (*c*) a brief alphabetical title index.

The collection is divided into ten primary classes, lettered A to K (omitting I), each of which is divided into sub-classes, numbered consecutively, and in some cases the sub-classes are further subdivided according to locality. Following the class-mark is the size-mark, which is indicated thus:—Colon (:) for works under 13 inches; slant rule (/) for works between 13 and 17 inches; parallel mark (||) for works over 17 inches wide. After the size-mark comes the work-mark; that is, the mark used to distinguish a given work from its fellows in a class.

The present list comprises 2563 works, distributed under 356 classes, and representing some 39,682 volumes.

Atoms and Energies. By D. A. Murray, A.M., sometime Instructor in the Government Shogyo Gakko Kyoto, Japan. London: Gay and Bird. 1901.

Starting with the assumption that the chemical atoms are all identical in substance, and accepting only two energies—one attraction or gravitation, and the other repulsion—the author considers in succession Chemical Affinity, Cohesion, Adhesion, Latent Heat, Magnetism, and Electricity, and attempts to give a rational explanation for these phenomena. His fundamental point is to regard the atoms of matter, not in the usual conception of small spherical particles, but as of very diverse shapes and sizes, and then with the two energies, gravitation and repulsion, to suggest that "all the so-called chemical potencies or attributes residing in the atoms, which differentiate them one from another, are simply the result of differences of shape."

The author shows ingenuity in his arguments; and what makes the book readable is the entire absence of that dogmatism so general in works of this kind; the ideas are merely put out as suggestions to "stimulate thought."

The final conclusion is, that "energy is a distinct entity," not a mode of motion; or, in other words, energy is that mysterious something which we have had to call Ether.

In an introductory chapter, Prof. Frederick Starr, of Chicago, speaks very highly of the work, considering the author's "point of view novel, the argument clear, and the book itself satisfactory."

It would scarcely be fair to the author in a notice of this kind to comment on any particular passage or point raised; the book must be studied as a whole, and the originality of the ideas certainly has the effect of stimulating thought and helps to guard one against the unfortunate habit of what Prof. Starr terms "giving some grand name to a difficult phenomenon and feeling about as well satisfied as if we had explained it."

CORRESPONDENCE.

ERRORS IN CHEMICAL TEXT-BOOKS.

To the Editor of the Chemical News.

Sir,—In Smith's translation of "Richter's Organic Chemistry" (vol. i., p. 240), I notice a very curious error in the description of some of my own work.

The reaction between sodium-ethyl and carbonic acid is properly set forth by the well-known equation, but there is the amusing statement, "This reaction is only applicable with sodium-methide and sodium-ethide."

In the *Jahresbericht* for the year 1868 (p. 523) there is an account of the preparation of caproate of soda by the action of sodium-amyl upon carbonic acid, and there can be no doubt whatever that the reaction is general and of the widest scope.

The action of sodium upon the compounds of zinc with the alcohol radicals takes place with great readiness, and the action of carbonic acid upon the resulting sodium compound is one of the quickest known to chemists. It is in the highest degree improbable that any term of the series of zinc organo-metallic compounds would fail to yield the corresponding sodium derivative, or that the sodium derivative would fail to react with carbonic acid.—I am, &c.,

J. Alfred Wanklyn.

The Laboratory, New Malden, May 26, 1902.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxiv., No. 18, May 5, 1902.

Glucose and Carbonates of Cerium. A New Method of Oxidation.—André Job.—The author examined the mechanism of the spontaneous oxidation of cerous carbonate dissolved in an alkaline liquor producing active oxygen. The carbonate of the peroxide which is formed from this in the cold by simple contact with air is an oxidising agent which allows of the formation of hydrogen peroxide when it is dissolved in acids. Recently M. Baur developed an application of these properties. By agitating with air an alkaline solution of cerous carbonate added to potassium arsenite, the oxidation of the latter is effected, and this fact verifies the assumption that the total quantity of oxygen fixed corresponds to the primary formation of the peroxide. The author continues these researches, and describes various experiments on the effect of glucose in causing the peroxidation of the cerium salt.

Alloys of Cadmium with Barium and Calcium.—Henri Gautier.—The author examines the conditions of formation and decomposition of the hydrides of the alkaline earths. It is not possible to produce these hydrides directly from the free metal, because, with the exception of calcium, they are not known in a state of purity. He therefore uses the alloys of the metals with an easily volatile metal, such as mercury or cadmium. The alloys of the alkaline earth metals with cadmium being easier to obtain pure and more concentrated than the corresponding amalgams, they are used by preference. He describes the preparation of the barium and calcium alloys, that of strontium having been already described.

An Oxycarbide of Cerium.—Jean Sterba.—The author obtains a well-defined and perfectly crystalline oxycarbide of cerium having a formula $CeC_2.2CeO_2$, which is always formed at the beginning of the reduction of the oxide by carbon. This oxycarbide is relatively stable in water and air. During its decomposition by acids it yields non-saturated hydrogen carbides. No other carbide except CeC_2 is formed in the electric furnace and an oxycarbide of formula $CeC_2.2CeO_2$.

Arsenic Anhydride and its Hydrates.—V. Auger.—The author performs a number of experiments on the effect of heat on $(AsO_4H_3)_2H_2O$. He finds that the only hydrates possible to obtain are $(AsO_4H_3)_2H_2O$ and $As_4O_{13}H_6$. The hydrates AsO_4H_3, $As_2O_7H_4$, and AsO_3H, described by Kopp, have not been obtained. Arsenic anhydride is produced at about 180°; it is stable even at 490°, but decomposes at a dull red heat, and cannot be obtained in a molten state.

Preparation and Properties of Chloro-, Bromo-, and Iodo-sulphobismuthates of Lead.—Fernand Ducalte. Sommerlad prepared certain sulphoarsenites and sulphoantimonites existing in nature by allowing a metallic chloride to act on arsenic or antimony sulphides. Cor-

sidering the chemical analogy of arsenic and antimony with bismuth, the author applies the same reaction to produce natural mineral sulphides of the latter metal. His researches as to the time of heating the mixture employed and the temperature to which it should be heated show that it is impossible to eliminate chlorine, but he succeeds in obtaining perfectly crystalline compounds of su'phobismuthates with chloro-, bromo-, and iodo-sulphides of bismuth. He therefore concludes that the presence of the halogen element in the products obtained by the action of lead chloride, bromide, or iodide on bismuth su'phide is not exceptional, and the proportion of the halogen element is determined by the general formula $PbS,B_2'S_3,2BiSX$, in which X can represent Cl, Br, or I.

Certain Derivatives of Pyruvyl-pyruvic Ether.—L. J. S mon.—In spite of the interest which attaches to its discovery very little is known of pyruvyl-pyruvic ether—

$$CH_3—CO—CO—CH_2—CO—CO_2C_2H_5.$$

During the action of aniline on ethyl-pyruvate the author isolates a compound—

$$CH_3—C(=N—C_6H_5)—CO—CH_2—C(=N—C_6H_5)—CO_2C_2H_5$$

By replacing the ethyl-pyruvate with another pyruvic ether and using paratoluidine in place of aniline, he obtains a series of compounds analogous to the preceding, and from a comparison of these he proposes the above formula.

Mutual Action of Acid Chlorides and Methanal—Marcel Descudé.—The action of an acid chloride on methanal may be expressed by the three following equations:—

a. $C_6H_5—COCl + CH_2O = C_6H_5—COOCH_2Cl.$

b. $\left.\begin{matrix}C_6H_5—COOCH_2Cl\\C_6H_5—COOCH_2Cl\end{matrix}\right\} = \left.\begin{matrix}C_6H_5—CO\\C_6H_5—CO\end{matrix}\right\rangle O + O\left\langle\begin{matrix}CH_2Cl\\CH_2Cl\end{matrix}\right.$

c. $\left.\begin{matrix}C_6H_5—CO\\C_6H_5—CO\end{matrix}\right\rangle O + CH_2O = \begin{matrix}C_6H_5—COO\\C_6H_5—COO\end{matrix}\rangle CH_2.$

Compounds of Tetrazoditolylsulphite of Sodium with the Aromatic Amines and the Phenols, and their Transformation into Azoic Colouring-matters.—MM. A. Seyewetz and Biot.—The authors find that the aromatic amines give with sodium tetrazoditolylsulphite compounds which can be transformed into azoic colouring matters by the action of light or by prolonged boiling with alcohol. They examine the theory of this transformation.

Addition of Hypochlorous Acid to Propylene.—Louis Henry.—This paper is an answer to Marc Tiffeneau's remark in a paper in the *Comptes Rendus* of April 7, 1902, on the subject of "The Constitution of the Chlorhydrines," in which he states that, contrary to Henry's assertion, during the fixation of ClOH on to propylene the oxyhydrile gives up to the carbon the least quantity of hydrogen, giving the chlorohydrine, $CH_3—CHOH—CH_2Cl$. The author now endeavours to re-establish his own statement.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xxv., No. 23.

Qualitative and Quantitative Estimation of Traces of Antimony in the presence of Large Quantities of Arsenic.—G. Denigès.—Already inserted.

Estimation of the Phosphoric Acid in Phosphates.—J. A. Muller.—Already inserted.

Estimation of Tin by Lenssen's Method.—J. A. Muller.—Already inserted.

Analysis of Tin Ores.—J. A. Muller.—Already inserted.

MEETINGS FOR THE WEEK.

MONDAY, June 2nd.—Royal Institution, 5. General Monthly Meeting.
—— Society of Chemical Industry, 8. "A Contribution to the Chemistry of Whiskey," by Dr. Philip Schridrowitz, F.C.S. "The Estimation of Perchlorate in Saltpetre, &c." by Dr. A. Dupré, M.A., F.R.S. "On the Will Test for Nitrocellulose," by Dr. Robert Robertson, M.A. "On the Effect of the Alcohol Duty on Chemical Industries," by Dr. O. Silberrad.

TUESDAY, 3rd.—Royal Institution, 3. "The Laws of Heredity, with special reference to Man," by Prof. Carl Pearson, M.A., F.R.S.

THURSDAY, 5th.—Royal Institution, 3. "Contemporary British Sculpture," by M. H. Spielmann.
—— Chemical, 8. "The Action of Ungerminated Barley Diastase on Starch" (Part I.), by J. L. Baker. "The Decomposition of Chlorates—Part V., Potassium Chlorate in presence of Oxides of Manganese," by W. H. Sodeau.

FRIDAY, 6th.—Royal Institution, 9. "The Nile Reservoir and Dams," by Sir Benjamin Baker, K.C.M.G., LL.D., D.Sc., F.R.S., M.Inst.C.E.

SATURDAY, 7th.—Royal Institution, 3. "The Development of the English Drama—III., The Drama under Elizabeth," by Prof. Brander Matthews, Litt.D., D.C.L.

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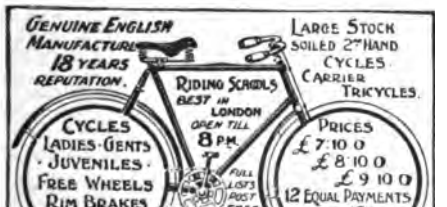
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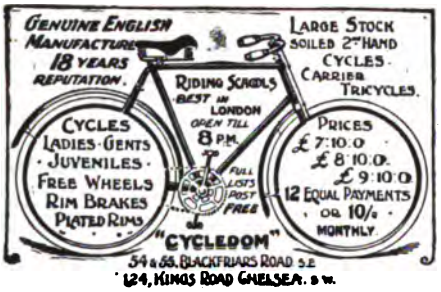
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THE CHEMICAL NEWS.

Vol. LXXXV., No. 2219.

CONTRIBUTIONS TO THE CHEMISTRY OF THE RARE EARTHS OF THE YTTRIUM GROUP.*

I.

By L. M. DENNIS and BENTON DALES.

(Continued from p. 258).

Historical (continued).

Soret (*Comptes Rendus*, lxxxvi., 1062), by a spectroscopic examination of Marignac's erbia and terbia materials, found a series of absorption bands which did not belong to erbia or to any other of the known elements. He called the earth giving them X. Cleve (*Comptes Rendus*, lxxxix., 478), in 1897, showed that by fractional decomposition of the nitrates by heat, erbia free from ytterbia and scandia could be separated into three earths, each characterised by an absorption spectrum which was a portion of the one ascribed to old erbia. The metals in these earths were the true erbium, with an atomic weight of about 166 (*Comptes Rendus*, xci., 381); holmium, and thulium with an atomic weight of about 170·7 (*Comptes Rendus*, xci., 328). Soret (*Comptes Rendus*, lxxxix., 521) stated that holmia and his earth X were indentical, and Cleve (*Comptes Rendus*, lxxxix., 708) admitted this to be true. Delafontaine maintained that his philippia was identical with both of these, but Cleve could not establish this fact. Delafontaine himself afterwards admitted that his philippia had no absorption spectrum. Lecoq de Boisbaudran (*Comptes Rendus*, cii., 1003, 1005) proved, by making many hundreds of fractions with ammonia and with potassium sulphate and alcohol, that the holmia spectrum could be divided into two portions, the bands characteristic of one being λ 640·4 and λ 536·3, and of the other λ 451·5 and λ 753. Since the first two bands were the ones by which Cleve and Soret originally characterised the element holmium, he kept this name for the one giving them; the one to which the other bands were due he called dysprosium. Crookes (*Proc. Roy. Soc.*, xl., 502) soon after Lecoq's announcement, stated that he had isolated an earth in this group giving only the absorption band λ 451, and that therefore dysprosium was probably still a complex substance. Lecoq has expressed the same opinion. Krüss and Nilson (*Ber. d. Chem. Ges.*, xx., 2134) showed, by a spectroscopic examination of the absorption bands of the rare earths obtained from many different minerals, that the variations in intensities of the bands as found in these minerals, could be accounted for only upon the assumption that the bands belonged to different individuals. As a result of this, they considered what we now call erbia to be made up of two components, thulia of two, holmia of four, and dysprosia of three. Hofmann and Krüss (*Zeit. Anorg. Chem.*, iii., 407) in 1893 concluded, as a result of their aniline hydrochloride fractionation, that holmium was a complex substance; Krüss (*Zeit. Anorg. Chem.*, iii., 353) alone has thrown doubt upon the individuality of erbia. By fractionation of his material rich in erbia from alcoholic solution with an alcoholic solution of aniline, then of the middle portions of this series as earth chloride aniline hydrochloride by ammonia, he could not get an earth whose base had a constant atomic weight. The beautiful absorption spectrum of erbia was discovered by Bahr in 1862 (*Sv. Vet. Akad. Handl.*, 1862, p. 597; *Liebig's Ann. Chem.*, cxxxi., 256). Crookes (Chem. News, liii., 75) mapped all of the spectra of erbia. The absorption, emission, and spark spectra of thulia have been studied by Thalén (Chem. News, xlvii., 217; *Comptes Rendus*, xci., 376).

In 1883 Crookes (*Phil. Trans. Roy. Soc.*, clxxiv., 891; Chem. News, xlvii., 261; xlix., 159, 169; 181, 194, 205) discovered that the anhydrous sulphates of certain rare earths became phosphorescent when exposed to the electric discharge in a vacuum tube, and that this light gave characteristic spectra. He (Chem. News, xliv., 23; *Proc. Roy. Soc.*, xxxii., 209) had in 1881 observed that some of the earths themselves phosphoresced under the same conditions. In 1883 he discovered in these phosphorescence spectra the citron band which he decided to be due to yttria. In 1884, Lecoq de Boisbaudran (*Comptes Rendus*, c., 1437) discovered that if the positive pole were immersed in a solution of some of the rare earths, and the negative one brought just above its surface, the light which was emitted on the passage of the spark gave an inversion spectrum which was nearly related to the phosphorescence spectrum of Crookes. Both were very delicate, and were greatly influenced by the presence of foreign oxides.

In 1886 and the year following Crookes (*Journ. Chem. Soc.*, lv., 250; Chem. News, liv., 115; lv., 83, 95) separated yttria by a large number of different fractionations into five portions, each of which had different phosphorescence spectra, but all of which showed the spark spectrum of yttria. The bodies responsible for these phosphoresence spectra he called "meta-elements," and he developed his theory of the genesis of the elements on the basis of this observation. In further support of the idea that yttria was a complex substance, he stated that Marignac's gadolinia gave him the same phosphorescence spectrum as yttria, except that the citron band was missing, and that the two green bands of samaria were present. Then a mixture of 61 parts of yttria with 39 of samaria gave the same phosphorescence as gadolinia, except for the presence of the citron band. He also found that the yttria from different minerals gave phosphorescence spectra with its rays of varying intensity, showing that a partial separation had taken place in nature.

This theory and all its conclusions were opposed by Lecoq de Boisbaudran (*Bull. Soc. Chim.*, [3], iii., 53). He (*Comptes Rendus*, cii., 1536) showed that pure yttria gave no phosphorescence phenomena, either by his method or by that of Crookes. According to him, the fluorescence bands were attributable to impurities, to his substances Z_α and Z_β, in fact, and these it was almost impossible to remove completely from the yttria (*Comptes Rendus*, ciii., 113). In connection with these substances giving fluorescence bands, he (*Comptes Rendus*, cv., 258, 301, 343, 784; cx., 24, 67) has studied a considerable number of fluorescences with well-defined spectral rays, having for solid solvents alumina, gallium oxide, silica, zirconia, stannic and tantalic oxides, and for active matter samaria and the oxides of Z_α and Z_β. Demarçay (*Rev. Gen. des Sci. Pures et Appliqués*, i., 396; Chem. News, lxii., 85) opposed Crookes's theory of "meta-elements," and stated that the radiant matter test of Crookes was far more delicate than the reversion spectra of Lecoq de Boisbaudran.

Dubois (*Comptes Rendus*, cvii., 99) made some compounds of yttria in the dry way. The accepted atomic weight of yttrium, 89·02 with O = 16 as the standard, is the one obtained by Cleve in 1882 (*Comptes Rendus*, xcv., 1225). Rowland (Chem. News, lxx., 68) published a method for the separation of yttria from other earths of its group by means of potassium ferrocyanide. His paper was severely criticised by Crookes (Chem. News, lxx., 81).

In 1896 Barrière (Chem. News, lxxiv., 159, 212) announced the existence, in the yttria earths from monazite, of a new metal which was separated from the other members of the group by precipitation with sodium thiosulphate in concentrated solution. This element, lucium, had an atomic weight of 104. Crookes (Chem. News, lxxiv., 259), by examination of its spark and absorption spectra, found that it was a mixture of yttrium and didymium, erbium and terbium, but was principally yttrium. Shapleigh (*Journ. Franklin Inst.*, cxliv., 68; Chem. News, lxxvi., 41) found by a quantitative analysis

* *Journal of the American Chemical Society*, vol. xxiv., No. 5.

of a sample of lucia that it contained 93·98 per cent of yttria earths, 3·74 per cent of ceria earths, 1·07 per cent of thoria, and 1·21 per cent of other foreign oxides.

In 1896 and 1897 Schützenberger and Boudouard (*Comptes Rendus*, cxxii., 697; cxxiii., 782; cxxvi., 1648) worked on the yttric earths of monazite sands. They found by fractional fusion of the nitrates and by fractional crystallisation of the sulphates that a lower limit of fractionation was reached, giving an earth whose metal-equivalent (R_2O_3) was about 95. They thought that they had found a new earth, whose metal had an atomic weight of about 102. Drossbach (*Ber. d. Chem. Ges.*, xxix., 2452), working also on monazite, thought he had a new earth similar to the others (the atomic weight of its base, 100). Further, Urbain and Budischovsky (*Comptes Rendus*, cxxiv., 618), fractioning the yttria earths by means of the acetyl acetonates, found a similar lower limit. Urbain (*Comptes Rendus*, cxxvi., 835; cxxvii., 107), working with the ethyl sulphates, succeeded in showing that this hypothetical earth could be separated into terbia ($R^{III} = 151·4$) at one end of his fractionation series and yttria ($R^{III} = 89$) at the other. In 1900 Urbain (*Ann. Chim. Phys.*, [7], xix., 184) again took up the question of this earth, and concluded that it was principally yttria, mixed with some erbia and terbia. This lower limit of various fractionation series, giving a base of nearly constant atomic weight, reminds one of the "oxide of gadolinium" of Nordenskjöld (*Comptes Rendus*, ciii., 795). This was a rare earth mixture, obtained from different minerals, which he knew to contain at least three oxides, those of yttrium, erbium, and terbium, and yet its metal had a constant atomic weight of 107.

Crookes (Chem. News, lxxviii., 134) in 1898 announced the existence of an element giving a group of phosphorescence bands in the ultra-violet only. He then called it monium. Its principal rays were λ 3120 and λ 3117, and its atomic weight about 118. In 1899, he renamed it victorium, and told how it was isolated (*Proc. Roy. Soc.*, lxv., 237; *Chem. Centrbl.*, 1899, II., p. 748). The method was a long combination of the standard ones of fusion of the yttria group nitrates, fractional precipitation of the oxalates from concentrated nitric acid solution, fusion of the nitrates again, and finally fractional precipitation with potassium sulphate.

In addition to all of these earths, and, of course, ceria, lanthana, didymia, and thoria, a few others have been announced. Their existence was generally short. Junonium was prepared in 1811 by Thomson from allanite (*Gilbert's Ann.*, xlii., 115; xliv., 113). Donarium was announced by Bergemann in 1851 (*Journ Prakt. Chem.*, liii., 239). He prepared it by precipitating the silica-free acid solution of the mineral thorite with ammonia. Damour (*Pogg. Ann.*, lxxxv., 555), Berlin (*Pogg. Ann.*, lxxxv., 556; lxxxvii., 608), and finally Bergemann himself (*Pogg. Ann.*, lxxxv., 558) thought that donaria was identical with thoria. Bahr (*Pogg. Ann.*, cxix., 572; *Journ. Prakt. Chem.*, xci., 179) announced the discovery of wasium, which Nicklès (*Comptes Rendus*, lvii., 740) thought was a mixture of yttrium with didymium or terbia. Delafontaine (*Liebig's Ann. Chem.*, cxxxi., 364) thought wasium was cerium, perhaps with some didymium. Popp (*Liebig's Ann. Chem.*, cxxxi., 364) agreed with Delafontaine. Bahr (*Liebig's Ann. Chem.*, cxxxii., 227) considered it probable that wasium and thorium were identical, and that both Nicklès and Delafontaine were mistaken in the matter.

Smith (*Nature*, xxi., 146; Chem. News, xlix., 182) announced the existence of two new elements in samarskite, which he called columbium and rogerium. This columbium is not, of course, to be confused with the well-known one. Chroustchoff (*Berg. u. Hust. Zeit.*, xlvi., 329; *Chem. Centrbl.*, 1887, p. 1277) detected spectroscopically the existence of a new element, russium, in certain wash residues from rocks, in many alumina preparations, and in American monazite. He (*Journ. Russ. Phys. Chem. Ges.*, xxix., 206; *Chem. Centrbl.*, 1897, xi., 329) prepared russium in much the same manner that Barrière prepared lucium. Linnemann (*Monatsh. Chem.*, vii., 121) discovered austrium in the rare earth mixtures from orthite. Lecoq de Boisbaudran (*Comptes Rendus*, cii., 1436) considered it to be gallium. Pribram (*Sitzungsber. d. k. Akad. d. Wiss., Wien*, Div. 2b, cix., 16; *Monatsh. Chem.*, xxi., 148) decided that Linnemann's product was gallium, at the same time maintaining the probability of a new element in orthite.

(To be continued).

NINETEENTH ANNUAL REPORT OF THE COMMITTEE ON INDEXING CHEMICAL LITERATURE.*

The Committee on Indexing Chemical Literature, appointed by your body in 1882, respectfully presents its Nineteenth Annual Report, embracing the fourteen months from June 1, 1900, to August 1, 1901.

Works Published.

"A Select Bibliography of Chemistry, 1492-1897." By Henry Carrington Bolton. Section VIII. Academic Dissertations. City of Washington: published by the Smithsonian Institution. 1901. 8vo. Pp. vi.+534.

This forms No. 1253 of the Smithsonian Miscellaneous Collections, vol. xli.

This Bibliography of Academic Dissertations is a second part of the work published in 1893, and with the First Supplement, issued in 1899, completes (if the term can be applied to bibliography) the undertaking begun by Dr. Bolton in 1888. The three volumes comprise about 25,000 titles. The dissertations found in the libraries of the Smithsonian Institution and of the U.S. Geological Survey are indicated by appropriate initials. There is a full subject index.

The completed manuscript of a "Bibliography of the Analytical Chemistry of Manganese," by Professor Henry P. Talbot and John W. Brown, has been carefully examined by your Committee, and they have recommended it for printing to the Smithsonian Institution, to which it has been presented.

Works in Progress.

Mr. Frank R. Fraprie reports progress on his manuscript "Index to the Literature of Cæsium, Rubidium, and Lithium," which is to be completed within a twelvemonth.

Mr. G. A. Smith, of Cornell University, reports that his "Index to the Literature of Selenium and Tellurium" will be completed by the close of the academic year.

Dr. M. D. Sohon is preparing for the press a "Subject-index to the Journal of the American Chemical Society."

Dr. H. Carrington Bolton has in preparation another "Supplement to the Select Bibliography of Chemistry," intended to cover the period beginning with 1897, and to include omissions.

Dr. Alfred Tuckerman has revised and prepared for the press the continuation of his "Index to the Literature of the Spectroscope"; the MS. has been presented to the Smithsonian Institution.

Notes.

During the past twelve months there have been published the following bibliographical works on chemical subjects:—

Bibliographia Lactaria. Bibliographie générale des travaux parus sur le lait et l'laitement jusqu'en 1899. Paris, 1900. 600 pp. 8vo.

Bulletin de la Société Chimique de Paris. Tables des années 1899 *a* 1898, *dressées par Th. Schneider.* Paris, 1900. Two parts.

* *Proceedings of the American Association for the Advancement of Science*, 1901, vol. l.

Zeitschrift für physikalische Chemie, Stöchiometrie, und Verwandschaftslehre. Namen und Sach-Register über Band i.—xxv., bearbeitet von T. Paul, Leipzig, 1900. 8vo.

And Professor A. K. Krupsky, of St. Petersburg, announces a 62-page "Bibliography of Chemistry" in Russian, which your Committee is as yet unable to describe more accurately.

Committee:—
H. Carrington Bolton (in Europe),
F. W. Clarke,
A. R. Leeds,
A. B. Prescott,
Alfred Tuckerman,
H. W. Wiley.

THE INFLUENCE OF THE WATER PRESENT ON THE FLASH-POINT AND COMBUSTION-POINT OF PETROLEUM.

By J. MATUSCHEK.

While examining a cloudy petroleum whose milky appearance was due to the presence of finely divided drops of water, I noticed with surprise that the flash-point, reduced to 760 m.m. pressure and 15° C., was between 23° and 24° C., and that the combustion-point was between 25° and 26° C. On shaking up 5 c.c. of this petroleum with an equal volume of sulphuric acid of 1·53 density the acid was observed to turn yellowish-brown, while a petroleum of good quality under the same conditions ought not to give more than a faint yellowish colouration.

It is well known that the presence of the bituminous products of distillation lowers both the flash-point and the combustion-point of a petroleum. We also know that in the presence of water the separation of the two liquids is fairly difficult, from which I have been led to the conclusion that the cause of high flash-points and combustion-points of petroleum was probably due to the proportion of water present.

To prove this supposition experimentally a petroleum was used whose density at 15° C. was 0·930.

The density of a petroleum of good quality should be from 0·79 to 0·82 at 15° C. When treated with sulphuric acid, as described above, this petroleum gave a brown colouration. On shaking up 5 c.c. of the petroleum with 2 c.c. of ammonia and a few drops of nitrate of silver, a brown colouration was produced, due to the reduction of silver by the sulphurised bituminous products. The flash-point and combustion-point, reduced to 760 m.m. and 15° C., were respectively 21° and 23° C. Fifty c.c. of this petroleum were then shaken up with 0·1 c.c. of distilled water until the mixture was homogeneous. The flash-point and combustion-point were then determined. The former was between 22° and 24° C., and the latter was 25° C.

Another 50 c.c. of the petroleum were then treated with 0·2 c.c. of water in the same manner, and the flash-point and combustion-point then obtained were respectively 27° C. and 29° C. With the addition of 0·3 c.c. of water 28° C. and 33·5° C. were obtained; 0·4 c.c. of water raised them to 30° C. and 34·5° C., 0·5 c.c. of water to 27° C. and 29° C., 0·6 c.c. of water to 28° C. and 30° C., and 0·7 c.c. of water to 30° C. and 32° C.

These facts show clearly that the proportion of water present in a petroleum contaminated with bituminous products of distillation raises the flash-point and the combustion-point of the petroleum; this increase depends on the quantity of water present.

The experiments show, further, that the regular rise of the flash-point and combustion-point is only maintained up to a certain degree; the regularity ceases as soon as small drops of water commence to form in the mixture during the experiment.

The formation of these small drops took place, in the last three experiments, within an hour at the ordinary temperature. After standing for twenty-four hours the first experiment only was still in the form of a homogeneous mixture.—*Œsterreich. Chem. Zeit.*, vol. iv., p. 209.

ANALYSIS OF TURF.

By H. BORNTRAGER.

Turf of recent formation is of a relatively light colour and small density; it differs considerably from heavy dark turf, which is often several centuries old. They both contain the same constituents, but in different proportions. For example, in 100 parts of turf dried at 100° there are found:—

	Fibre.	Humic acid.	Nitrogen.
Light turf	95—90	5—10	0·5—1·0
Heavy turf	58—48	40—50	2·0—2·5

Light turf absorbs a large quantity of water, while heavy turf hardly absorbs any at all. The French methods of analysis take no notice of any difference between fibre and humic acid, and the author is of the opinion that it is important to estimate these substances separately when dealing with a turf required for agriculture or distillation.

In an analysis of turf destined for these uses the following are determined:—

1. *Moisture.*—A finely divided portion is dried at 100° until the weight is constant. The quantity varies between 10 and 40 per cent.
2. *Mineral Wax.*—The dried turf is extracted with anhydrous ether; the results vary from 0·5 to 1·0 per cent.
3. *Nitrogen.*—By Kjeldahl's method we generally find from 0·5 to 2·5 per cent. About half the nitrogen is present in the form of ammonia, which can be estimated by boiling 1 part of the turf with a concentrated solution of caustic soda, and collecting the ammonia given off in titrated acid.
4. *Humic Acid and Fibre.*—One or 2 grms. are extracted three times by boiling for an hour with 5 grms. of soda and 200 grms. of water; the extracts are filtered on a tared filter. After washing, the residue is dried at 105°; this represents the fibre. The humic acid is then estimated in the alkaline filtrate.
5. *Ash.*—About 1 grm. is incinerated with nitrate of ammonium in a platinum crucible without a cover. The proportion varies from 2 to 10 per cent.

French analyses give the principal constituents of the ash; that is to say, the insoluble matter, the lime, magnesia, potash, and phosphoric acid; but in the author's opinion these estimations are of little importance. There is also a rapid method for the estimation of humic acid alone, already described by the author and M. Tacke (*Chem. Zeit.*, 1896, p. 23; and 1893, p. 174). A known weight of turf is boiled with a finely powdered carbonate, the carbonic acid given off is estimated and the quantity of humic acid present is deduced by calculation.

The following are typical analyses made by the author's method:—

	Light turf from Hanover.		Heavy turf from Aldenbourg.	
	I.	II.	III.	IV.
Water	30·0	29·0	20·0	20·0
Ash	3·0	3·1	2·8	3·0
Fibre	55·0	54·9	49·0	47·0
Humic acid	12·0	13·0	38·0	30·0
Total	100·0	100·0	109·0	100·0
Total nitrogen	1·1	1·3	1·8	2·0
Nitrogen as ammonia	0·3	0·4	0·6	0·7

Turf intended for use as fuel should naturally have its calorimetric power estimated.—*Zeit. Anal. Chem.*, 1901.

ANALYSIS OF COMMERCIAL CYANIDES.

An Exact Method for the Determination of Cyanic Acid; a Double Cyanide; and an Antidote for Cyanides.

By O. HERTIG.

The use of cyanide of potassium and cyanide of sodium for the extraction of gold is constantly increasing. The attention of manufacturers is therefore directed to the most economical methods possible for the preparation of these salts, and to all industries in which they are obtained as by-products—I need only instance the manufacture of lighting gas. Analysts have also been engaged in devising more exact and more rapid methods for the estimation of cyanides.

First of all, there is M. Mellor's work (*Zeit. Anal. Chem.*, vol. xl., p. 17) on the determination of cyanides and cyanates. The author recommends nitrate of calcium to precipitate the carbonates which are always present in small quantities. Nitrate of barium, recommended by Holdenhauer (Lunge, "Chem. Tech. Untersuchungsmethoden," vol. i., p. 438), is rejected; cyanate of barium is, in fact, insoluble in water. In so far as the determination of cyanogen in cyanides is concerned, M. Denigès's method, recommended by Mellor, is certainly the best and most convenient. The method of Fordos and Gelis (Lunge, *loc. cit.*, vol. i., p. 489) always gives too high results. But the method of Mellor and Allen for the determination of cyanic acid cannot be approved of. This titration with nitric acid, followed by a back-titration with soda, gives very different results to those obtained by other methods. It seems that the temperature at which the mixture of cyanide and cyanate of silver is to be digested, 50° (for how long?), is not high enough. It would appear to be preferable to let the nitric acid act on the cyanate of silver for an hour at the temperature of the water-bath. It must also be remembered that the filtrate which is titrated back with soda contains an ammoniacal salt, according to the equation—

$$AgOCN+2HNO_3+H_2O=AgNO_3+NH_4NO_3+CO_2.$$

Still, analyses I have made do not enable me yet to pronounce a definite opinion on this method.

The method of estimation I propose is based on a determination of the nitrogen, and on the decomposition of the cyanates by hydrochloric or sulphuric acid. This decomposition gives rise to an ammoniacal salt, as shown in the following equations:—

$$KOCN+2HCl+H_2O = KCl+NH_4Cl+CO_2.$$
$$2KOCN+2H_2SO_4+2H_2O = (NH_4)_2SO_4+K_2SO_4+2CO_2.$$

To determine the cyanic acid in the cyanides we take 0·2 to 0·5 grm. of material. This is dissolved in a few c.c. of water, and treated in a porcelain crucible with dilute hydrochloric or sulphuric acid; it is then evaporated to dryness on the water-bath and taken up with water. The ammonia is estimated in this solution by boiling with soda and collecting the gas which escapes in 1/5th N sulphuric acid. After the operation the acid is re-titrated with 1/5th N ammonia, using fluorescein as an indicator. The presence of chloride of ammonium may lead to errors if we estimate the potassium with chloride of platinum after having decomposed the cyanates with hydrochloric acid. When we wish to estimate the potassium, it is necessary, after decomposing the cyanates by hydrochloric acid in a platinum crucible, to drive off the chloride of ammonium by calcination. It must not be heated beyond a dull red, or there would be a risk of decomposing the chloride of potassium.

I have recently had a sample of cyanide containing 38·85 per cent of cyanogen (pure cyanide of potassium contains 40 per cent of CN and 60 per cent K). Now, the proportion of potassium was 31·45 per cent, and that of the nitrogen 15·83 per cent.

The complete analysis gave the following results:—

	Per cent.
Insoluble part (SiO_2, Al_2O_3, Fe_2O_3) ..	0·03
NH_4	0·33
K	19·23
CN	31·45
CO_2	38·85
CNO	2·94
O (calculating CO_2 into K_2CO_3)	2·65
OH	2·93
Cl	0·44
Total	99·91

The question was, acting on these results, to determine the composition of the substance. By treating a sample of the material with warm alcohol, a residue of carbonate of potassium was obtained. The proportion of OH, obtained by nitrate of magnesium, could only be in combination with the potash, and not the ammonia. It must be admitted also that the cyanic acid was united with the potash, as the ammoniacal solutions of this acid decompose rapidly, while the solution of the substance under examination remained for several days without change. From the above it would appear that we had to deal with a double cyanide of potassium and ammonium of the composition:—

	Per cent.
Insoluble part	0·03
Moisture	0·33
NH_4CN	46·98
KCN	27·72
K_2CO_3	9·22
KCNO	5·06
KOH (corresponding to 2·93 per cent OH, instead of 3·16 per cent)	9·65
KCl	0·92
	99·91

I ought to say that the figure 3·16 per cent was obtained with the nitrate of magnesium method; this figure is rather too high. The figure 2·93 was found by calculating the potassium remaining into potash.

This double cyanide of potassium and ammonium occurs in pure white amorphous fragments having a strong odour of hydrocyanic acid. When calcined in an open crucible, it increases in weight; this is due to the formation of a cyanate. Its aqueous solution remains unchanged for several days, and no azulinic acid is formed ($C_4H_5N_5O$).

It cannot be decided with any certainty whether this salt is suitable for the extraction of gold from tailings without making experiments on an extended scale. Neither can I describe the manner of preparation of this compound.

The question of an antidote for hydrocyanic acid is always of importance, considering how the use of cyanides is increasing. It has been solved by M. Robert and his pupil, M. Krohl. On this point I would call attention to "Merck's Annual Report for the Year 1899"; it recommends the use of peroxide of hydrogen as an antidote for cyanides. A 30 per cent solution is administered for internal use, and a 3 per cent solution for subcutaneous injections.

Peroxide of hydrogen transforms hydrocyanic acid into a harmless oxamide:—

$$2CNH+H_2O_2 = \begin{matrix}CONH_2\\ |\\ CONH_2\end{matrix}.$$

For the past three years this method has been used successfully in cases of poisoning by cyanide in English mining districts (*Journal of the Chemical and Metallurgical Society of South Africa*, 1899). It would be advisable, therefore, to insert this treatment by peroxide of hydrogen in the various publications which are met with under the general title of "First Aid to the Injured." In

the greater number of these cases of poisoning, it was only inhalation of prussic acid fumes which had occurred, and every chemist or foreman can easily learn how to administer a subcutaneous injection.—*Zeit. für Angew. Chemie*, 1901, p. 619.

ON A MODIFICATION OF ROSE'S METHOD OF SEPARATING COBALT AND NICKEL.*

By R. L. TAYLOR, F.C.S.

NUMEROUS methods have been proposed for the purpose of separating cobalt and nickel when in solution together. The two metals, however, are so much alike in their general properties, that very few methods appear to give satisfactory results, and those few are, as a rule, troublesome and tedious.

The method which I have described as "Rose's method" was proposed by him over fifty years ago (*Pogg. Ann.*, Bd. 71, p. 545), and a translation of Rose's paper, by T. H. Henry, appeared in the *Chemical Gazette* for 1847, page 362, accompanied by some remarks on the method by the translator. The process, while fairly satisfactory, was very tedious and troublesome, and probably for that reason, it appears to have gone out of use. It is described in "Gmelin's Handbook," and in various editions of Fresenius's "Quantitative Analysis" but very few modern text-books even mention the reaction (which is interesting and important) upon which the method depends. For example, it is not referred to in Roscoe and Schorlemmer's "Treatise on Chemistry," and there is no mention of it in the newer Watts' "Dictionary of Chemistry."

Rose's method depends upon the fact that cobalt is precipitated as the black sesquioxide by various insoluble carbonates (such as those of barium, strontium, and calcium) in presence of free chlorine or bromine, and that nickel is not so precipitated. The following equation practically represents what takes place in the case of cobalt:—$2CoCl_2+3BaCO_3+Cl_2=Co_2O_3+3BaCl_2+3CO_2$. It is very probable, however, that the cobalt is converted from a cobaltous to a cobaltic salt before it is precipitated by the carbonate.

According to his original description of the process, Rose passed a stream of chlorine through a somewhat dilute solution of the chlorides of the two metals, containing a considerable excess of hydrochloric acid, for several hours. He then added excess of barium carbonate and allowed to stand, with frequent agitation, for from twelve to eighteen hours. By the end of that time the cobalt was all converted into sesquioxide, and the nickel remained in solution, and could be filtered off. In his remarks on Rose's method (referred to above) Henry suggested the use of bromine instead of chlorine. In a subsequent letter (*Chem. Gaz.*, 1855, p. 237), Henry stated that he had, by keeping the temperature at 120° F., succeeded in reducing the time required to a few hours. He considered that the results, however, were not so good as those obtained by Liebig's method of separation by means of potassium cyanide.

Induced principally by a desire to find a better method than those usually employed for the qualitative separation and detection of nickel in presence of cobalt, I have recently made some experiments on the action of barium and calcium carbonate on solutions of the two metals, in presence of chlorine and bromine. As the result of these experiments, I find that the method of Rose can be considerably shortened, with the result also of making it much more accurate.

The mistake which both Rose and Henry made was in the use of free hydrochloric acid before the addition of chlorine (or bromine) and the carbonate. Rose mentions having tried a neutral solution, but with unsatisfactory results. On the other hand, I find that if there is *no* free acid present, barium or calcium carbonate, in presence of chlorine or bromine, will precipitate cobalt completely in three or four minutes. Perhaps barium carbonate acts a little more rapidly than calcium carbonate, but, in a great many experiments I have made, neither of them has ever failed to precipitate the cobalt in five minutes.

With a neutral solution of nickel, on the other hand, I have never found any precipitation of the black oxide within an hour. After a longer time, a black deposit begins to form on the side of the vessel at the edge of the liquid, where the air has access to it. If air is excluded nickel scarcely appears to be precipitated at all. I have had solutions of nickel in presence of both barium and calcium carbonate and bromine water in stoppered bottles for some weeks without any appearance of precipitation of the black oxide. When the liquid is heated to a temperature of 70° or 80° C., the nickel is rapidly precipitated as the black sesquioxide.

If, however, much free acid is present when the carbonate and bromine water are added, as was always the case in following Rose's original method, the precipitation of the cobalt is very considerably retarded. It may take hours, and sometimes does not even begin for over half-an-hour. This explains why Rose found it necessary to allow his solutions to stand so many hours.

The retarding effect of the free acid is rather surprising, because all free acid naturally becomes neutralised by the carbonate employed, which must always be in excess. My first impression was that the soluble barium or calcium salt formed when the acid is neutralised by the carbonate must be the cause of the retardation, but experiment showed that it was not. The cause of the retardation is simply free carbonic acid. If, after adding the carbonate to a solution of cobalt containing free acid, the liquid is boiled (to expel the carbon dioxide) and cooled again before adding the bromine water, there is no retardation. Also, if carbon dioxide is bubbled through a *neutral* solution of cobalt, or if such a solution is mixed with ordinary soda-water, on adding the carbonate and bromine water there is a retardation of the precipitation of the cobalt similar to that observed when free acid is present at the outset.

This curious retarding action of free carbonic acid appears additionally strange when it is borne in mind that carbon dioxide is one of the products of the reaction itself, and that immediately the reaction begins there must be free carbonic acid present in the liquid. Plainly, the retarding action of the free carbonic acid is very slight unless there is a considerable amount of it in proportion to the water, and in dilute solutions the amount never becomes sufficient. In *strong* neutral solutions the carbonic acid produced during the reaction has its effect, and the precipitation of the cobalt takes much longer. At present I am unable to suggest any reason why free carbonic acid should exert this curious retarding effect.*

With a dilute solution which is approximately neutral the process of separation works rapidly and well. I have tested it quantitatively with most satisfactory results. The materials I used were the crystallised chlorides of the two metals, each guaranteed by the makers as free from the other.

A solution of chloride of nickel was prepared, of no definite strength, and the amount of nickel in it was determined by electrolytic deposition, from an ammoniacal solution, on a weighed platinum basin—a process of estimating nickel which appears to be most satisfactory. In this way, in two separate experiments 50 c.c. of the solution gave 0·1350 and 0·1355 grms. of metallic nickel respectively. In two further experiments, 50 c.c. of the same solution were mixed, in each case, with an indefinite

* A Paper read before the Manchester Literary and Philosophical Society, February 18, 1902.

* It is well known that manganese is precipitated by barium or calcium carbonate under the same circumstances as cobalt, and I find that free carbonic acid acts exactly as it does with cobalt—the precipitation of the manganese is very considerably retarded.

amount of a solution of cobalt. The two metals were then separated by adding excess of precipitated barium carbonate, made into a paste with water, and bromine water. Ten minutes were allowed for the precipitation of the cobalt. After filtering and washing, the barium was removed from the filtrate by precipitation with sulphuric acid, the liquid evaporated to a small bulk, and the nickel determined by electrolytic deposition. The two experiments gave respectively 0·1345 and 0·1347 grms. of nickel.

In a similar way a solution of chloride of cobalt was prepared, and the amount of cobalt in it determined by electrolytic deposition from a solution of the double oxalate of cobalt and ammonium, this being one of the solutions of cobalt which is said to give the best results. But, in my hands at any rate, the process of electrolytic deposition is not so satisfactory with cobalt as with nickel. The deposited metal is not so clean, and it is difficult to know when the process is complete. Thus, in one experiment 50 c.c. of the solution of cobalt, electrolysed for five hours (when the deposition seemed to be complete), gave 0·1517 grm. of metallic cobalt. In another experiment, when the electrolysis was carried on through the night (altogether about seventeen hours) 50 c.c. gave 0·1537 grm. of cobalt. Possibly the larger amount represents the true strength of the solution.

Mixtures were next made of 50 c.c. of each of the two solutions, the separation effected as before, and both the cobalt and nickel determined electrolytically. In one experiment, where the electrolysis of the cobalt solution was allowed to proceed throughout the night, 0·1542 grm. of cobalt and 0·1352 grm. of nickel were obtained. In another, where the cobalt solution was electrolysed for seven hours, the numbers were 0·1532 and 0·1347 respectively.

In addition to the above results obtained by myself, some experiments on this method of separation have been made by Mr. David Segaller, A.R.C.S., Dublin, with equally satisfactory results.

The above results are very much better than those quoted by Rose and Henry for the original method. This may be due to the fact that my materials were probably purer than any they were able to obtain. The results demonstrate quite satisfactorily that by this method the separation of cobalt and nickel is not only rapid, but complete.

The process not only acts well quantitatively, but I recommend it very strongly for the separation and detection of cobalt and nickel in the ordinary process of qualitative analysis. The mixed sulphides of cobalt and nickel are dissolved in the usual way in dilute hydrochloric acid with the aid of a crystal of potassium chlorate. The liquid is then boiled down just to dryness in order to expel the free acid. The residue is taken up with water, and precipitated barium or calcium carbonate and bromine water added.* It is now allowed to stand for five minutes, with frequent shaking. If cobalt is present, a black precipitate very soon appears. At the end of five minutes the liquid is filtered, and the filtrate tested for nickel by the addition of a drop or two of ammonia and ammonium sulphide. The presence of cobalt in the precipitate may be confirmed by the borax bead test. In this way nickel may be detected even when present in very small quantity, and when the cobalt is largely in excess.

Instead of boiling off the free acid, it may be neutralised with sodium or potassium hydrate before adding the carbonate and bromine water; or the carbonate of barium or calcium may be added in excess to the acid liquid, and then the liquid boiled for a short time to expel the free carbon dioxide. It must then be cooled to the ordinary temperature before adding the bromine water.

The carbonates of barium, strontinum, and calcium are not the only ones which act in the manner described; the carbonates of magnesium and zinc apparently act in the same way. Thomas Moore (CHEM. NEWS, 1900, vol. lxxxii., p. 73), describes the use of zinc oxide and bromine water for the purpose of estimating small quantities of cobalt in presence of nickel. Mr. Moore finds that those reagents precipitate cobalt as sesquioxide, but not nickel, even on boiling. He insists that bromine *must* be used, and not chlorine. I have tried the experiment, and fail to find any difference at all between the action of chlorine and bromine.

* Barium and calcium carbonates appear to act equally well. The dry substances may be employed. Barium carbonate is preferable if the subsequent removal of the added metal is desired.

ZIRCONS FROM CEYLON.

ILLUSTRATING RANGE AND CHANGE OF DENSITY AND COLOUR.

Exhibited by Prof. A. H. CHURCH, D.Sc., F.R.S., at the Royal Society Conversazione, May 14, 1902.

ZIRCONS, GREEN.—Range of density, 4·0 to 4·19; raised by heating from 4·0 to 4·31, from 4·11 to 4·51, from 4·19 to 4·39, and from 4·19 to 4·57. These almost alone amongst zircons emit a brilliant orange light during grinding with diamond dust. This phenomenon does not occur with these stones after their density has been raised by strong heating.

ZIRCONS, GOLDEN.—Range of density, 4·38 to 4·45; raised by heating to 4·66. These zircons continuously glow with an orange incandescence in the flame of a Bunsen burner.

ZIRCONS, YELLOW.—Range of density, 4·6 to 4·64; raised by heating to 4·7, the colour being sometimes lost completely.

ZIRCONS, RED.—Range of density, 4·67 to 4·71, unaltered by heating, but the colour reduced.

The molecular volume of zirconia, if normal, would correspond to a density in zircon of 4·0, and would then be in accord with the molecular volumes of the other bin oxides belonging to Mendeleeff's Group IV.

The contraction suffered by certain zircons when strongly heated was first observed by Henneberg in 1846. It was more fully studied by Damour in 1864 and by the Exhibitor in the same year. In one of the instances shown it corresponds to a reduction of volume from 100 to 91·3.

The striking absorption spectrum of certain zircons is shown in a careful drawing by Dr. C. A. MacMunn. This spectrum (first observed and published by the Exhibitor in 1866) has not before been accurately mapped, although Becquerel gave the wave-lengths of the several bands in 1888. Dr. MacMunn's measurements for the darkest parts of eleven bands are:—

λ	λ
689·5	589·0
683·5 (not shown in the drawing)	562·0
661·0	538·0
653·0	515·0
622·5	484·0
616·0	

Tellurium-diphenyl and the Atomic Weight of Tellurium.—O. Steiner.—The author first prepared telluride of phenyl according to the method of MM. Krafft and Lyons, by heating 36·5 grms. of pure powdered tellurium with 50 grms. of mercury-diphenyl in a sealed exhausted tube at a temperature of 220—230° for fifteen hours:—$(C_6H_5)_2Hg + 2Te = (C_6H_5)_2Te + HgTe$. The contents of the tube are exhausted with ether and filtered to separate the HgTe, and a brown oil is collected; this can be rectified at a pressure of 100 m.m. Thus an oil is obtained which is again heated for three hours at 200° with 5 grms. of powdered tellurium. This is again rectified *in vacuo*, and then once more over copper turnings. Finally, a highly refracting oil is obtained, boiling at 174° and 100 m.m., and at 110—112° *in vacuo*. The analysis of this product, $(C_6H_5)_2Te$, carried out on different portions, give for O = 16 at the ordinary pressure, Te = 126·4 to 126·7.—*Berichte*, vol. xxxiv., p. 570.

THE

RADIO-ACTIVITY OF THORIUM COMPOUNDS.

I. An Investigation of the Radio-active Emanation.

By E. RUTHERFORD, M.A., D.Sc.,
Macdonald Professor of Physics, McGill University, Montreal,
and
FREDERICK SODDY, B.A. (Oxon.),
Demonstrator in Chemistry, McGill University, Montreal.

The following paper contains a preliminary account of an investigation into the property possessed by the compounds of thorium of giving a radio-active emanation, and also into the nature of the emanation itself.

It was shown by one of us (*Phil. Mag.*, 1900, [5], xlix., 1, 161) that the compounds of thorium, besides being radio-active in the same sense as the uranium compounds, also continuously emit into the surrounding atmosphere, under ordinary conditions, something which, whatever its real nature may be, behaves in all respects like a radio-active gas. This "emanation," as it has been named, is the source of rays, which, like the Röntgen and uranium rays, and the ordinary well recognised type of thorium radiation, will darken a photographic plate, and will render a gas capable of conducting an electric current (that is, will "ionise" it), but is sharply distinguished from them by the following considerations. It can be moved from the neighbourhood of the thorium compound by a current of air passing over it, or even by the process of ordinary gaseous diffusion, and transported long distances, so that the characteristic photographic and ionisation effects appear in the air far away from the original source of radio-activity. The Röntgen and uranium rays, as is well known, travel in straight lines from their source, and any object opaque to them interposed in their path will sharply screen the space behind. But in the case of the thorium radiation there is no such screening effect, because here we have a case of a substance emitting, not only straight line radiation, but also particles of a gas, itself radio-active, capable of diffusing through the surrounding atmosphere around obstacles placed in its direct path, and so arriving and producing its effects at points completely screened from rays travelling from the thorium in straight lines. It was shown in the original communication that these effects could not be ascribed to minute particles of thoria dust carried off mechanically, and all the subsequent work on the subject shows that the hypothesis that the compounds of thorium emit a radio-active gas is not merely the only one which will explain the facts, but that it does so in every observed case in a completely satisfactory manner.

Present State of the Subject from a Physical Standpoint.

In the papers referred to, the general character of the phenomena in question was presented, and a short *résumé* will perhaps not be out of place here. It was shown that the radiation from the emanation decays rapidly, but at a perfectly defined rate; that is, the effects it produces diminish with the lapse of time, falling to about one-half the original value at the end of one minute. This "rate of decay," as will be shown later, is of great value in identifying and distinguishing between different types of emanation.

The emanation passes unchanged through cotton-wool, weak and strong sulphuric acid, and aluminium and other metals in the form of foil, but not through an extremely thin sheet of mica.

The emanating power of thoria is independent of the surrounding atmosphere, but is destroyed to a large extent by intense ignition, and does not return when the substance is kept.

One of the most striking properties of the thorium emanation is its power of exciting radio-activity on all surfaces with which it comes in contact; that is, a substance after being exposed for some time in the presence of the emanation behaves as if it were covered with an invisible layer of an intensely radio-active material. If the thoria is exposed in a strong electric field, the excited radio-activity is entirely confined to the negatively charged surface. In this way, it is possible to concentrate the excited radio-activity on a very small area. The excited radio-activity itself has a regular rate of decay, but different from that of the emanation, its effect falling to half value in about eleven hours. There is a very close connection between the excited radio-activity and the emanation. It was shown that the amount of the former produced under various conditions was proportional to the amount of the latter, and if the emanating power of thoria be destroyed by intense ignition, its power to excite radio-activity correspondingly disappears. Some apparent discrepancies which at first stood in the way of too close a connection being inferred have resolved themselves by recent work into strong confirmation of the view that the two are related to each other as cause and effect.

Another remarkable property of the excited radio-activity is that it is soluble in sulphuric and hydrochloric acids; that is, a platinum wire, rendered radio-active by being made the negative pole of an electric field in the neighbourhood of some thoria, will give up its radio-activity to these acids. If the acid be then evaporated, the radio-activity remains on the dish, whilst if left to itself the radio-activity of the acid solution decays at a rate identical with that of the original excited radio-activity on the platinum wire.

Simultaneously with the discovery of excited radio-activity due to thoria, Curie showed that radio-active barium possessed a similar property. Later, Dorn (*Abh. der Naturforsch. Ges. für Halle-a-S.*, 1900) repeated the work quoted for thoria, and extended it to include two preparations of radio-active barium compounds (radium) prepared by P. de Haen, and a preparation of radio-active bismuth (polonium). He found that radium gave out an emanation which was similar to that from thoria, but which retained its radio-active power much longer. The excited radio-activity from radium, on the other hand, decayed more rapidly than that from thoria. The special property of emitting an emanation is, however, confined to the compounds of radium and thorium, those of uranium and polonium do not possess it to an appreciable extent.

An approximate determination of the molecular weight of the emanation produced by radium has been carried out (Rutherford and H. T. Brooks, *Nature*, 1901, lxiv., 157) by a diffusion method, taking advantage of the slow rate of decay of the radium emanation. From comparison of the rate of diffusion of gases of known molecular weight into one another, it was found that the molecular weight probably lies between 40 and 100.

It seemed probable that an examination of the phenomena by chemical methods might throw light upon its nature, and the emanation produced by thoria was chosen as more suitable for the purpose than that produced by radium, on account of the obscurity still surrounding the chemistry of the latter, and the difficulty of producing material of even approximate uniformity of properties. Thoria, on the other hand, is an article of commerce, and specimens from different sources show surprising uniformity in this respect.

During the progress of the work, the subject has acquired additional importance and interest through the discovery by Elster and Geitel (*Phys. Zeit.*, 1901, ii., 590) that it is possible to produce excited radio-activity from the atmosphere, without further agency, by simply exposing a wire highly charged to a negative potential in the atmosphere for many hours, and that this also possesses the property of being dissolved off by acids, and of being left behind unchanged on the evaporation of the latter. But here again the rate of decay is different from that of the excited radio-activity produced by thoria, which is evidence for assuming that the two are probably not identical, although so strikingly analogous. However, the

close connection between excited radio-activity and the emanation established in the case of thoria renders it probable that the excited radio-activity obtained from the atmosphere is caused by the presence there of an emanation or radio-active gas analogous to, although probably different from, the thorium emanation. The discovery is likely, as Elster and Geitel point out, to have important bearings on the theory of atmospheric electricity, and in our opinion renders a close study of the thorium emanation the more imperative.

(To be continued).

PROCEEDINGS OF SOCIETIES.

PHYSICAL SOCIETY.

Ordinary Meeting, May 23rd, 1902.

Prof. S. P. Thompson, President, in the Chair.

Mr. T. C. Porter showed A Lecture Experiment on the Ebullition of Rotating Water.

If the water in a beaker, having approximately vertical sides, be caused to rotate about an axis concentric with the vertical geometrical axis of the beaker, it is obvious that in any horizontal section of the water the pressure is least in the centre and increases from the centre outwards. If the temperature of the water is just below boiling-point and heat is supplied to it whilst it is rotating, steam is formed only in the region of least pressure, and a gaseous core is produced. The rotation can be given to the water by stirring it with a glass rod covered with a piece of india-rubber tubing, and maintaining the stirring motion during the act of withdrawal of the rod. Some curious phenomena are shown by the column of steam, if the water is first stirred and then left to come to rest whilst the heating is continued. At first there is a markedly concave surface to the water in the beaker, and the column of steam is practically continuous from base to summit. After this stage pulsations set in. The course of events during a single pulsation is as follows:—1st phase, the surface of the water flattens, and when most nearly level a column of steam springs up with great rapidity from the base of the beaker to the surface of the water, heaving this up in its central portion. Immediately after the eruption of the steam, and whilst the column still stretches from the base of the beaker to the surface of the water, follows the second phase. The steam column seems to condense, and breaks up, leaving only a few small bubbles, which either hang stationary or move downwards in the liquid; whilst if the water has dust in it, the motion of the dust particles shows that a curious kind of annular wave, concentric with the steam column, traverses the water from top to bottom, apparently causing in its course the almost complete condensation of the steam, and the curious brief downward movement of the bubbles left. At the same time the surface of the water in the beaker becomes deeply indented. After this the apex of the surface vortex rises, and the first phase of the phenomenon recurs. The curve of the surface of the water throughout is never a parabola, the divergence indicating that the water rotates more rapidly as it nears the axis. The pulsations can also be produced by stirring cold water in a beaker-shaped jar, having a small hole in its bottom through which a stream of air-bubbles can be blown. The forms of the steam columns in some cases present a likeness to those of solar prominences; and Mr. Porter suggested that the immediate cause of the latter might be the diminution of pressure on the sun's surface at, or near, the centre or centres of depressions caused by violent cyclonic disturbances in the solar atmosphere. He asked for an explanation of the pulsations.

Mr. C. V. Boys exhibited a small Heat Engine in which rotating water evolved steam without ebullition.

Mr. T. H. Blakesley expressed his interest in the apparent want of buoyancy of the steam-bubbles in the second phase of the pulsation. He said that in filtering liquids he had sometimes noticed what appeared to be large bubbles of air which lacked buoyancy. They were really drops of the liquid surrounded by a film of air. He suggested that the curve of the water surface could be made parabolic by rotating the beaker. In this case the vertical distance between the highest and lowest points of the surface would be a measure of the velocity of rotation.

Mr. W. Watson said that as the viscosity of water changed rapidly when nearing the boiling-point, small variations in the temperature of the liquid might alter the parabolic form of the surface. Any bubbles of steam formed away from the axis of rotation would be driven to the axis on account of the greater density of the water.

Mr. Price said he had seen some similar experiments illustrating the formation of water spouts. The appearance was like the effect shown, but the pulsations were absent.

Dr. A. Griffiths, in suggesting an explanation of the oscillation of the water, pointed out that if the distance of a rotating body (held in position by a spring) from the axis be forcibly diminished, the conservation of the moment of momentum indicates an increased rate of rotation. On releasing the body, the increased centrifugal force causes it to shoot past its original position. Beyond the position of equilibrium, the conservation of the moment of momentum indicates a diminished speed which decreases the centrifugal force. Thus the body oscillates radially. When the water in the beaker is flat, the mass of the water is at a less average distance from the axis of rotation than when it is heaped towards the sides. Thus the outward-driving force is probably larger when the depression is small than when it is large. The outward force drives the water beyond the position of equilibrium which it would hold in the absence of oscillation.

The Chairman said it would be interesting to observe the effects produced by varying the method of supplying the heat to the water.

A paper by Mr. J. A. Erskine, on "*The Conservation of Entropy*," was read by the Secretary.

Heat energy may be expressed as the product of two factors—a quantity factor, entropy, and an intensity factor, temperature. The conservation of entropy holds in thermodynamics when dealing with reversible processes, and is analogous to the conservation of other quantity factors such as momentum, moment of momentum, and electric quantity. The author shows the completeness of the analogies by considering Carnot cycles carried out on electrostatic and hydraulic engines. Prof. Wiedeburg has proposed to extend the doctrine of the conservation of entropy to irreversible processes by introducing a new quantity analogous to electric resistance.

The Secretary read a letter from Mr. Swinburne, in which he stated that the author had got hold of what was no doubt Clausius's original idea of entropy. The science of thermodynamics, as far as it deals with reversible processes, depended upon what the author called the conservation of entropy, but reversible processes did not exist in practice. The author's treatment was quite in accordance with orthodox thermodynamics.

A paper by Sig. G. Giorgi, on "*Rational Units of Electromagnetism*," was read by Mr. Price.

Mr. Price prefaced the reading of the paper by saying that both Prof. Fleming and Prof. Fessenden had advocated a partial change of units which would leave the most important ones unchanged, and the method employed by the author was similar to that adopted by Prof. Fessenden. The author starts with a set of three equations, which contain explicitly the four concrete units of E.M.F., M.M.F., electric current, and magnetic current, together with that of activity, and considers them as fundamental in electromagnetism. Two fundamental units are required to express these quantities, and their product must reproduce the mechanical unit of activity. If the watt is assumed as

unit of activity, there are two units ready made, the volt and the ampère, which satisfy the condition, and may be considered fundamental. All concrete units in electricity and magnetism can be expressed in terms of these and the second as unit of time. In order to complete the system, a unit of length is required. The metre and kilogramme are consistent with the watt, and putting them together with the units enumerated in the paper, the author has built up an absolute metre-kilogramme-second system which comprises electric, magnetic, and mechanical measures in a consistent frame.

The Secretary read a letter from Mr. Swinburne stating that most physicists who love simplicity would be glad to get rid of "4π," but schemes for suppressing it generally moved it somewhere else. He asked if the author had really got rid of 4π or merely chained it up in another place. If a change in units were to be made, it would be an advantage to get rid of the terms electromotive force and magnetomotive force.

Prof. Everett said he had arrived at the following determinations of the author's meaning:—

When μ and K are taken into account, we have the two following identities:—

1. The dimensions of current are the same as those of magnetomotive force, or H × length, called f in the paper.
2. The dimensions of electromotive force are the same as those of quantity of magnetism divided by time —called by the author magnetic current, and denoted by g.

In connection with 1, the line-integral of H for a path singly linked with a circuit carrying a current of C ampères is to be called C. This gives the unit for H in terms of the ampère and the unit of length (the metre).

In connection with 2, we are to use the equation for magnetoelectric induction in a circuit

$$\text{electromotive force} = -\frac{dN}{dt},$$

together with the rule that a quantity of the magnetism m gives the total flux $N = m$. The unit quantity of magnetism is thus defined in terms of the volt and the unit of time (the second).

The Chairman said he had received a paper from Sig. Giorgi, giving a number of illustrations of the application of his theory to calculations. There were also some further explanations, and the dimensions of many physical quantities were tabulated in terms of the volt, ampère, and second. The author claimed to have based an absolute system on the metre, kilogramme, and second, while leaving the practical units unchanged.

The Society then adjourned until June 13th.

The Preparation of Chlorine by means of Chlorate of Sodium, and the Preparation of Trichloride of Phosphorus.—C. Graebe.—To prepare chlorine, hydrochloric acid, D = 1·1 (or on the large scale D = 1·2), is heated to incipient boiling, and by the aid of a stoppered funnel with a long thin stem a fine stream of commercial chlorate of sodium at 500 grms. per litre is run in. The gas is thus given off at about 100°, and only contains very small quantities of peroxide of chlorine, which in this proportion is without objection for the ordinary uses of chlorine. It can, however, be removed if so desired by means of a wash-bottle, ClO_2 being more soluble in water than chlorine is. To prepare the trichloride of phosphorus, chlorine is passed into a flask containing red phosphorus under a layer of PCl_3 prepared previously. The tube carrying the chlorine plunges into the PCl_3 close to the phosphorus, and is arranged in such a manner that by the aid of a stirrer the end can be cleared if it becomes obstructed by a deposit of PCl_5. The flask is connected by a tube to a vertical condenser. The flask is then heated gently so that the PCl_5 may react on the excess of red phosphorus; finally, it is distilled, and the product is obtained free from free phosphorus.—*Berichte*, xxxiv., p. 645.

NOTICES OF BOOKS.

Water Supply. Considered Principally from a Sanitary Standpoint. By William P. Mason. Third Edition, Re-written. New York: John Wiley and Sons. London: Chapman and Hall, Lim. 1902. Pp. vii.-448. 8vo. Illustrated. Cloth.

The author of this valuable work might have used the following lines by way of introduction:—

> "Traverse the desert, and then ye can tell
> What treasures exist in the cold deep well
> Sink in despair on the red parch'd earth,
> And then ye may reckon what water is worth."

since it deals with the value of normal water, the insidious dangers of polluted water, the relations of disease to drinking waters, methods for the artificial purification of water, as by filter-beds on a large scale, rain, ice, and snow as sources of water, surface and deep-seated waters, and water statistics.

The opening chapter, which is short, contains some account of the magnitude of ancient water supplies, with especial reference to Rome; the second chapter considers the question of the wholesomeness of water, and cites striking instances of dangerous epidemics of cholera and typhoid disseminated by means of drinking-water. The different conditions in Hamburg and in Altona during the terrible outbreak of cholera in 1892 are clearly described and the circumstances leading to the prevalence of typhoid fever in certain localities are made known; on the whole, the author finds that it is not a simple or easy matter to define "good, pure, wholesome water." The artificial purification of water by mechanical filtration as carried on in England and in Germany and the American Filter System are detailed in Chapter III., the descriptions being facilitated by the aid of diagrams, plans, and photographic views. The author has studied systems of water supply in many parts of Europe; he describes in an interesting way the underground storage systems of Naples, and of Gibraltar, where he enjoyed unusual opportunities.

This third edition has been practically re-written, and the chapters in earlier editions on the chemical and bacteriological examination of water have been omitted, appearing independently in book-form. The sources of information are scrupulously acknowledged. The volume is embellished by handsome illustrations, and its utility is enhanced by a very full index. H. C. B.

The Principles of Inorganic Chemistry. By Wilhelm Ostwald. Translated with the Author's sanction by Alexander Findlay, M.A., B.Sc., Ph.D. With 122 Figures in the Text. London: Macmillan and Co., Ltd. New York: The Macmillan Company. 1902. Pp. 785.

This translation of the "Grundlinien der Anorganischen Chemie" will doubtless contribute to a more widespread knowledge of the recent developments of general chemistry, and their importance in the study of the other branches of the science.

The work, which is divided into no fewer than forty-four chapters, begins with a discussion of general principles, the laws of conservation, the phenomena of combustion, &c.; it then deals seriatim, chapter after chapter, with all the known chemical elements, giving first a few general remarks on their characteristics, properties, appearance, &c.; then their compounds are dealt with, not only from the chemical, but from the physical point of view; this is more especially the case with regard to gases, their liquefaction and solidification.

The general intention of the author has been to make a book of *pure* chemistry; regard has been paid to the related sciences and arts, only in so far as chemical questions play a part in them; by this means the student will obtain a thorough "grounding" in chemistry, and will find it easier to apply his knowledge in later years.

The book is well printed and extensively indexed.

with the separation of CrO_3, otherwise stable on contact with water, contrary to the corresponding salts of potassium and ammonium. At the other end of the series the basic chromate, $2Na_2O.CrO_3+13H_2O$, is found in large pale yellow flakes, very soluble, and fusible at about 50°. The neutral chromate occurs with $10H_2O$, with $4H_2O$, and also anhydrous. The second of these hydrates has no analogue in the series of sulphates of sodium; it is formed between 20° and 65°.—*Berichte*, vol. xxxiii., p. 3686.

The Alkaline-cupric Carbonates.—M. Gröger.—A concentrated solution of bicarbonate of potassium saturated with carbonic acid, being treated progressively in the cold with a solution of sulphate of copper, gives a deep blue liquor, which, after several days, deposits silky needles of a greenish-blue colour, decomposed by distilled water, giving a basic carbonate of copper and bicarbonate of potassium. These needles have the composition—

$$2CO_3K_2.8CuO.7CO_2.17H_2O$$

or— $$4CO_3KH.8CuO.5CO_2.15H_2O.$$

Deville (*Ann. Chim. Phys.*, Series 3, vol. xxix., p. 102) has described a carbonate of the composition $CO_3K_2.5CuO.4CO_2.10H_2O$. This salt was obtained in the presence of CO_3K_2, and might be mixed with a little basic carbonate of copper; it is probably identical with that of the author. Things happen differently in the case of bicarbonate of soda; we only obtain a definite product in the presence of the neutral carbonate. If we dissolve 20 grms. of CO_3Na_2 and 50 grms. of CO_3NaH in 700 c.c. of water, and add 8 grms. of SO_4Cu+5H_2O dissolved in 20 c.c. of water, we find that the filtered liquid deposits, in one night, pale blue tufts of a neutral sodio-cupric carbonate with the very simple formula $CO_3Na_2.CO_3Cu.3H_2O$. The deposit of this salt, like that of the preceding ones, is accelerated in the presence of a solid alkaline bicarbonate, and the crystals are then less needle-like.—*Berichte*, vol. xxxix., p. 429.

NOTES AND QUERIES.

*** Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

River Water.—I should be much obliged if any of your readers could give me any particulars of analyses of river water from the Argentine Republic, Chili, or any of the South American States.—Ernest A. Lewis.

Iodine and Starch Test-paper.—Will you be good enough to inform me if it is possible by any method or chemical to render permanent the colour produced by the presence of iodine on chemical test-paper composed of starch-paste. When iodine is present, this paper assumes a blue colour, or blue marks are produced, which fade or disappear in the course of a day or two, but which I desire to make permanent.—Chemical.

MEETINGS FOR THE WEEK.

Friday, 13th.—Royal Institution, 9. "The Progress of Electric Space Telegraphy," by G. Marconi.

—— Physical, 3 30. (At the National Physical Laboratory, Bushy House, Teddington).

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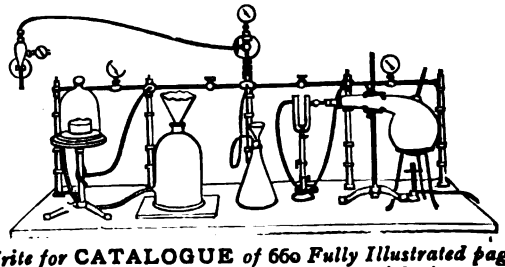

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COEFFICIENTS OF THE CUBICAL EXPANSION OF ICE, HYDRATED SALTS, SOLID CARBONIC ACID, AND OTHER SUBSTANCES AT LOW TEMPERATURES.*

By JAMES DEWAR, M.A., D.Sc, LL.D.,
Jacksonian Professor, Cambridge University, and Fullerian, Royal Institution, London.

The apparent specific gravities of boiling liquid oxygen which resulted from weighing in the liquid a series of metals and other substances were given in a lecture entitled "New Researches on Liquid Air," printed in the Royal Institution *Proceedings* for 1896. For instance, silver, calc-spar, rock crystal, and iodide of silver gave the respective apparent densities 1·1278, 1·1352, 1·1316, and 1·1372. On correcting the weight of liquid displaced by each substance for contraction to −182·6°—by calculating a Fizeau mean coefficient of expansion for the range of temperature employed, on the assumption that the parabolic formula might be legitimately extended to low temperatures—it was found that the real density of liquid oxygen so deduced for all the bodies used was, as a mean, 1·137.

The determination of the densities of substances at the temperature of the boiling-point of oxygen—and hence of their mean coefficients of expansion between that temperature and ordinary temperatures—opens out a very large field of investigation, from which, if a sufficiently large number of observations were available, valuable deductions might be drawn. On account, however, of the expense and trouble of producing quantities of liquid oxygen, its use for this purpose is not likely to become general, although, when available, it is the easiest body to use in conducting such experiments, especially when the vacuum vessel containing it is immersed in a larger vessel containing the same fluid or well evaporated air. The ease with which liquid air can now be obtained in many laboratories suggests that its application to work of this kind would in some cases be a convenience, and the present investigation was undertaken with the desire of ascertaining what accuracy could be attained, and how the method could be applied to inorganic or organic substances which occur in the form of fine crystals. The use of a mixture of varying composition and density like liquid air necessitates a determination of its density with accuracy and rapidity before and during the course of the experiments. For this purpose, in the experiments about to be detailed, the liquid air that had been allowed to evaporate for twenty-four hours in advance was used in large silver-coated vacuum vessels of some 3 litres capacity. In order to ascertain the density of the liquid, a polished silver ball, which had been weighed once for all in liquid oxygen, was weighed in the sample of liquid air, and from the relative weights thus found the density of the liquid air could be approximately determined, assuming that of liquid oxygen to be 1·137.† To prevent any disturbing ebullition in the liquid-air flask in which the weighings took place, and to reduce the rate of its evaporation to a minimum during the course of an experiment, the substance to be used was previously cooled in a supplementary vessel containing liquid air, and then transferred to the large flask. To avoid as far as possible the formation of cracks in the bodies during the process of immersion in the liquid air, it was found advisable to cool them slowly in the air of the vacuum flask first, and then to lower them into the liquid. In this way, with proper care and attention, results were obtained comparable in accuracy with the density taken in liquid oxygen. Substances like solid carbonic acid and ice were weighed in the cool, gaseous air of the vacuum vessel, and their weights subsequently corrected for buoyancy. The temperature of the densest and lightest samples of liquid air were ascertained by the hydrogen thermometer, and that of the others deduced by graphic interpolation. As the entire range of temperature through which the bodies were cooled amounted to about 200°, a degree or two up or down has no real influence on the results; the extreme range of temperature in the air samples was from 83·8° to 86·1° Abs.

When the body to be examined was a salt, it was employed in the form of a compressed block. One experiment was, however, made in a section of a large crystal of chrome-alum. The salt, previously reduced to a fine powder, was moistened with water and compressed in a cylindrical steel mould under great hydraulic pressure. During compression the saturated salt solution drained away, and finally a cylindrical block of some 50 grms. of the salt was obtained free from porosity and hard enough to allow its surface to be polished. In this form salts and other materials similarly treated are especially adapted for accurate specific gravity determinations. After such treatment it was found that all the mechanically attached water was got rid of in the case of hydrated salts, and also in such as did not combine with water. In order to get cylindrical blocks of the salts showing no porosity, the presence of water, or rather the saturated salt solution, was found to be essential during the application of pressure. In the same way it was found to be an advantage in compressing such a substance as solid carbonic acid, to moisten it with a fluid like ether before applying the hydraulic pressure.

Recalling the work of Playfair and Joule ("Researches on Atomic Volume and Specific Gravity," *Chem. Soc. Journ.*, vol. i., 121), which originated in a suggestion of Dalton's that the volume of a hydrated salt in solution was simply the volume of the water of crystallisation, ice and some hydrated salts were selected, as well as some other bodies whose coefficients of expansion they had determined. Substances of special interest were included in the list, like mercury, sulphur, iodine, and solid carbonic acid, the latter being particularly important as an example of a solidified gas.

In the further conduct of an experiment the observations made on a substance were three, namely:—(A) the weight in grammes of the substance and suspending platinum wire, either in air of about 17° C. temperature or in the gaseous air in the flask containing the liquid air; (B) the weight in grammes of the body and wire when immersed in the liquid air; and (C) the weight in grammes of the suspending platinum wire in ordinary (17°) air. This wire was bent into the form of a small cage, to hold the compressed cylinder or portion of the substance to be examined. (C) was measured once for all for each cage, and in the different cases varied from about 0·2 to 0·3 grms. The relative portion of wire not immersed in liquid air was so small that no appreciable error will be made in (B) by considering that the whole of the wire was immersed. Further, no allowance is made for the (17°) air displaced by the weights themselves. The observed weights A, B, C are therefore connected with the weights and specific gravities of the substances concerned by the very approximate relations.

$$A = W + w - \frac{W}{S}d - \frac{w}{s}d,$$

$$B = W + w - \frac{W}{S}D - \frac{w}{s}D,$$

$$C = \quad w \quad - \frac{w}{s}d.$$

* A Paper read before the Royal Society, May 1, 1902.

† As the correction due to the contraction of the silver ball between the temperature of boiling oxygen and that of the air sample is small, it may be neglected.

where W = true weight of substance examined.
w = true weight of platinum wire.
S = specific gravity of substance required.
D = specific gravity of liquid air, when B was observed.
d = specific gravity of gaseous air, when A was observed.
s = specific gravity of platinum.

From these relations we derive—

$$S = \frac{A - C - B\frac{d}{D} + C\frac{d}{D}\frac{s-D}{s-d}}{A - B - C\frac{D-d}{s-d}}D,$$

instead of which we may write—

$$S = \frac{A-C}{A-B}D \times \text{correcting factor.}$$

The correcting factor is—

$$\frac{1 - \frac{d}{D}\frac{B-C\frac{s-D}{s-d}}{A-C}}{1 - \frac{D-d}{s-d}\frac{C}{A-B}},$$

which in the present circumstances differs from unity only by a few units on 10,000.

In the subjoined Table I. are given the values of A − C, A − B, and D for each observation.

In the case of substances of less density than liquid air, a polished copper ball weighing 38 grms. was used as a sinker. The details of the ice experiments, three in number, are given in Table II.

Two experiments were made on compressed cylinders of solid carbonic acid. In the first of these the carbonic acid was compressed dry, in the second after a few drops of ether were added. The specific gravities of solid mercury, iodine, and sulphur were also determined in liquid air. The iodine was in the form of a compressed cylinder, but the sulphur was a piece of a crystalline mass of native origin.

The specific gravity of the actual portion of the substance weighed in the liquid air was, with one or two exceptions, determined also at the temperature of the laboratory, about 17° C. From the two sets of observations the value of the mean coefficient of cubical expansion between 17° C., and the temperature of liquid air, was calculated.

In calculating coefficients of expansion, various forms may be given to the formula employed, and correspondingly different results may be obtained from the same set of observations. For short ranges of temperature these results are practically identical, but this no longer holds for a range of temperature such as we have in these experiments. All that is possible in the present instance is to adopt a linear formula. The usual formula is $v_T = v_0(1 + \alpha T)$, where the value v_0 at 0° C. becomes v_T at T° C. when α is the coefficient of expansion. If we use densities (d) instead of volumes (v) this formula becomes—

$$d_0 = d_T(1 + \alpha T), \quad \text{or} \quad \alpha = \frac{d_0 - d_T}{T d_T}; \quad \alpha = 0{\cdot}000538.$$

Another formula, when T and T′ are the temperatures dealt with, is—

$$d_T = d_{T'}\{1 + \alpha(T' - T)\}, \text{or} \quad \alpha = \frac{d_T - d_{T'}}{(T' - T)d_{T'}}; \quad \alpha = 0{\cdot}000595.$$

Again—

$$d_{T'} = d_T\{1 - \alpha(T' - T)\}, \text{or} \quad \alpha = \frac{d_T - d_{T'}}{(T' - T)d_T}; \quad \alpha = 0{\cdot}000558.$$

Also we may choose a mean formula—

$$\alpha = \frac{d_T - d_{T'}}{(T' - T)\frac{d_T + d_{T'}}{2}}; \quad \alpha = 0{\cdot}000576.$$

The differences in the results of applying these formulæ are shown in the numerical values attached to each, which are calculated from the first experiment on solid carbonic acid in Table III., coupled with the specific gravity 1·53 of the solid at −78° C.

Perhaps as a matter of general convenience, the first of these formulæ is the best; however, the second was chosen to conform with the old work of Playfair and Joule, and it is the results of this formula which appear in the table.

The temperature range is taken from about −186° C. to 17° C., unless otherwise stated.

Ice.—In determining the density at the temperature of liquid air of pieces of clear ice cut from large blocks, both the silver and copper balls already referred to were used as indicated. The true weight *in vacuo* of the silver ball was 132·2855 grms., and that of the copper ball was 38·0802 grms. The observations and results are given in Table II. The mean of the three densities at −188·7° C. is 0·92999.

Recently Vincent (*Roy. Soc. Proc.*, 1901) has re-determined the density of artificial ice at the freezing-point, and also its coefficient of expansion. He finds the density to be 0·916, or from his tabulated results 0·91599. Playfair and Joule find the mean of the densities given by eight observers previous to them, to be 0·919, and they themselves get 0·9184; Bunsen found it to be 0·9167. If we take this most recent determination, namely, 0·91599 at 0°, and 0·92999 at −188·7°, and use the formula—

$$d_0 = d_T(1 + \alpha T)$$

we get α = 0·00008099.

Vincent refers to "only one" estimate for natural ice, namely, 0·0001125, adding that "the mean of three available results for artificial ice is 0·000160"; finally, he gives the mean of four determinations of his own, namely, 0·000152. Apparently then, we may take 0·0001551 as the mean coefficient of expansion of ice between 0° and (say) −20° C. Thus the mean coefficient of expansion between 0° and −188° C. is about half of that between 0° and −20° C. The mean coefficient of expansion of water in passing from 4° to −10° is −0·000362, and from 4° to 40° C. it is 0·00002155. Hence the mean coefficient of expansion of ice between 0° and −188° C., is about one-fourth of that of water between 0° and −10° C., and half of that between 4° and 100° C.

If we had the densities of ice at still lower temperatures, the values of the coefficient of expansion thence determined would, we have every reason to believe, be less than what we have found. We shall therefore not be overstraining the argument if we use the value just found to determine an upper limit to the density of ice at the absolute zero. The result is 0·9368, corresponding to a specific volume 1·0675. Now the lowest density of water, namely, at the boiling-point, is 0·9586 (corresponding to specific volume 1·0432), so that ice can never be cooled low enough to reduce its volume to that of the liquid taken at any temperature under one atmosphere pressure. In other words, ice molecules can never be so closely packed by thermal contraction as the water molecules are in the liquid condition, or the volume of ice at the absolute zero is not the minimum volume of the water molecules. It has been observed by Professor Poynting ("Change of State, Solid, Liquid," *Phil. Mag.*, 1881) that if we suppose water could be cooled without freezing, then taking Brunner's coefficient for ice, and Hallstrom's formula for the volume of water at temperatures below 4° C., it follows that ice and water would have the same specific volume at some temperature between −120° and −130°; applying the ordinary thermodynamic relation, then no change of state between ice and water could be brought about below this temperature. On the other hand, Clausius ("Mechanical Theory of Heat," 1879, p. 172) has shown that the latent heat of fusion of ice must be lowered with the temperature of fusion some 0·603 of a unit per degree. If such a decrement is

assumed to be constant, then about −130° the latent heat of fluidity would vanish.* Baynes discusses the same subject ("Lessons on Thermodynamics," 1878, p. 169), and arrives at the conclusion that at a temperature of −122·8° C. and under a pressure of 16,632 atmospheres there is no distinction between the solid and liquid forms of water. At temperatures below this limit no amount of pressure would transform ice into water. We are thus relieved from a difficulty that would follow but for this demonstration of Clausius, namely, that the application of enormous pressures to ice, even at temperatures below that of liquid hydrogen, might cause the transformation of ice into water.

Carbonic Acid.—Two experiments were made with this substance, the masses in each case being about 20 grms. These were compressed cylinders; the former was compressed dry, while the latter was slightened moistened with ether. The data and results are given in Table III.

The density of solid carbonic acid at its boiling-point was formally given as 1·5 (see *Proc. Roy. Inst.*, 1878, "The Liquefaction of Gases"), but the mean of my results at the time came to 1·53. Recently the same value has been found by Behn. Taking this value and 1·6267, the mean of the above results at −188·8° C., and using the formula $d_T = d_{T'}\{1 + \alpha(T' - T)\}$ we get $\alpha = 0{\cdot}0005704$.

This is a very large coefficient of expansion, being greater than that of any substance recorded in Table I., and comparable with that of sulphur between 80° and 100°, which, according to Kopp, is 0·00062. The coefficient of liquid carbonic acid at its melting-point taken from the recent observations of Behn (*Chem. Journ.*, 1901) is 0·002989, so that the rate of expansion of the liquid at its smallest value is very nearly five times that of the solid.

Solid Mercury.—One experiment was made with solid mercury, of which the details are given in Table I.

Mallet determined with great accuracy the density of solid mercury at −38·85°, his result being 14·193; coupling this with the density found for the liquid-air temperature, we find the value of the coefficient of expansion between the melting-point and −189° C. is 0·0000887. For fluid mercury above 0° C. the mean value is about 0·000182, so that in the solid state this coefficient is about half of that in the fluid state.

(To be continued).

ON THE PREPARATION OF TANTALUM BY THE ELECTRIC FURNACE, AND ON ITS PROPERTIES.

By HENRI MOISSAN.

In a previous paper it has been shown that niobite reduced to powder and mixed with sugar charcoal, gives, in the electric furnace, a button rich in niobium and in tantalum. The metallic mass, treated with hydrofluoric acid containing a little nitric acid, then by fluoride of potassium, gave a mixture of fluotantalate and fluoxyniobate of potassium. On applying Marignac's method of separation to these salts we were able to prepare about 4 kilogrms. of fluoxyniobate and fluotantalate of potassium.

The mixture of these two salts was exhausted six times with boiling water. The fluotantalate remaining, after having been washed with distilled water, was decomposed with its own weight of sulphuric acid in a platinum crucible. The white precipitate, carefully washed with warm water, was calcined. The tantalic acid still contained 0·32 per cent of sulphur in the form of sulphuric acid in combination, after washing and calcination. Ten per cent of pure carbonate of ammonium were mixed with this tantalic acid and calcined. This operation was repeated twice.

After having made sure of the purity of the tantalic acid, by means of its density and by its molecular weight, we attempted to reduce it by means of carbon at the temperature of the electric furnace.

We recalled the fact that Berzelius by heating the fluotantalate of potassium with potassium obtained a greyish powder which did not conduct electricity, and had a density of 10·08. Henri Rose, by decomposing the fluotantalate of sodium by metallic sodium, obtained a black powder with a density of 10·78, and which was, further, a good conductor of electricity.

Children, by the electrolysis of tantalic acid with an intense current, obtained a tantalum with a reddish-yellow colour and which was very brittle.

Finally, Marignac, by decomposing a fluotantalate with aluminium, prepared and described a crystallised alloy of tantalum and aluminium.

Tantalic acid has been regarded up to the present as being infusible and irreducible by carbon. We have treated this tantalic acid with a quantity of sugar carbon corresponding to the reaction $Ta_2O_5 + 5C = 2Ta + 5CO$.

To obtain a button containing only a small amount of carbon it is advisable to increase the proportion of tantalic acid by one-tenth.

The mixture was agglomerated by pressure into the form of small cylinders, which were heated in a Perrot furnace in a brasque of sugar charcoal. After this calcination, which gives a certain consistency to the mixture, the cylinders are arranged in a graphite crucible, which is heated in the electric furnace. The cylinders may also be placed in a graphite boat in the centre of a tube formed of the same substance. In both cases the temperature used should be very high if we wish to melt the resulting ingot of tantalum. If this is not done, though the reduction is properly effected, the cylinders, while fritting a little, still keep their shape approximately, and have under a lens a cavernous metallic appearance, and are covered with a yellow or orange layer. After heating for five minutes with a current of 800 ampères and 60 volts only a fritted mass is obtained; it is necessary to heat for ten minutes to effect the fusion of the tantalum.

Properties of Tantalum.

This tantalum when it is sufficiently heated occurs in the form of a brilliant metallic mass with a crystalline fracture. Certain preparations have given us an ingot containing no more than 0·5 per cent of carbon, but in this case more than half the tantalum was volatilised during the operation. This sample, containing such a small quantity of carbon, scratches glass and rock-crystal with ease; it is infusible before the oxyhydrogen blow-pipe, which transforms it rapidly into tantalic acid. It can be fused in the electric furnace, but only with a very powerful arc. Its density has been found to be 12·79, while the density of the tantalum prepared by Berzelius was 10·08, and that of Rose's tantalum 10·78.

Finely powdered tantalum takes fire when gently heated in fluorine, and gives off abundant vapours which, on condensation on a cold body, form a fluoride.

In a current of chlorine the attack of tantalum commences slowly at 150°. It increases at about 200°, and finally at 250° the combination takes place with incandescence; a chloride of tantalum is sublimed in the form of long orange-yellow needles. This chloride is easily fusible, and volatilises with decomposition in an atmosphere of chlorine. Bromine can be distilled over powdered tantalum without producing any reaction, but at a dull red heat it commences to give a yellow sublimate, and at the temperature of the softening of glass the reaction becomes complete with the disengagement of a large quantity of heat.

The vapour of iodine does not react on tantalum, even at a temperature of 600°.

* In my paper "On the Lowering of the Freezing-point of Water by Pressure" (*Roy. Soc. Proc.*, 1880), it was proved that up to 700 atmospheres the rate of fall was constant and equal to the theoretical value within the range of pressure if the difference between the specific volumes of ice and water remain constant; thence the latent heat of fusion must diminish just as Clausius had predicted.

If we place some tantalum in a current of dry oxygen it is necessary to raise it to a temperature of 600° (taken with a thermo-electric couple) before it takes fire, when it continues to burn with lively incandescence.

At a temperature of 700° sulphur, selenium, and tellurium do not react. When heated in a current of nitrogen at 1200° tantalum only increases very slightly in weight, and is not transformed into a nitride. Phosphorus and arsenic may be distilled over tantalum at the temperature of the softening of glass without producing any reaction. The same is the case with antimony.

An ingot of tantalum containing only 0·6 per cent of carbon did not combine with sodium, potassium, or zinc in fusion; when heated with iron in the electric furnace a small portion entered into combination, but the ingot did not show any homogeneity.

Hydrochloric acid gas attacks tantalum, giving a white sublimate, which becomes darker in colour as the temperature rises; at the same time hydrogen is given off. Water vapour and sulphuretted hydrogen do not react on tantalum at a temperature of 600°, or if any reaction does occur it is entirely superficial.

Ammonia-gas is decomposed at a dull red heat by powdered tantalum, of which the colour deepens without any change of weight. Sulphurous acid gas is reduced by tantalum with incandescence towards 500°, producing an abundant deposit of sulphur and an oxide of tantalum. At the same temperature nitrous and nitric oxides react with lively incandescence. Phosphoric anhydride is easily reduced above a red heat, giving off abundant fumes of phosphorus, but without incandescence.

Powdered tantalum gently heated with iodic anhydride becomes incandescent, and gives off abundant vapours of iodine. Arsenic acid is reduced by tantalum at above a red heat.

The compounds rich in oxygen, such as binoxide of manganese, are reduced by tantalum with incandescence.

Protoxide and binoxide of lead gently heated with powdered tantalum become incandescent, and leave a residue consisting of a black sponge containing small globules of melted lead. The protochloride and bichloride of mercury are easily reduced when heated, and give a sublimate of metallic mercury.

Fused potash is attacked by tantalum; it gives off hydrogen, and forms an alkaline salt. Heated chlorate of potash does not react on tantalum, even at its temperature of decomposition. We have already shown that the same was not the case with niobium. This is therefore one of the reactions which differentiates these two simple bodies.

The attack of tantalum by nitrate of potassium in fusion also proceeds more slowly than with niobium; there is no incandescence, and peroxide of nitrogen is given off.

Acids alone are without action on tantalum, with this exception—that boiling concentrated sulphuric acid attacks it very slowly, taking a brown colour. Aqua regia is without action, but in the same manner as with silicon and niobium, tantalum is easily attacked by a mixture of nitric and hydrofluoric acids.

These different reactions thus establish the fact that tantalum possesses very particular reducing powers, which cause it to resemble the metalloids rather than the metals.

Its reactions are similar to those of niobium. This parallelism is very pronounced, and the principal difference between these two simple bodies is that niobium is a more energetic reducing agent than tantalum.

Analysis.

The estimation of the carbon was made by combustion. A weighed quantity of the metal reduced to powder was heated in a boat in a current of oxygen. The whole of the carbon is transformed into carbonic acid, which is collected and weighed. From the increase in the weight of the boat we can calculate the amount of tantalic acid formed and the amount of tantalum originally present.

Care must be taken in this analysis to make quite certain that the tantalum has been completely transformed into tantalic acid.

By this means we have found the following figures, taking 183 as the atomic weight of tantalum:—

	I.	II.	III.	IV.	V.
Combined carbon ..	2·5	1·8	1·1	0·66	0·5
Tantalum	96·7	97·35	—	—	—

—*Bull. Soc. Chim.*, Series 3, vol. xxvii., No. 10.

THE ELECTROLYTIC SEPARATION OF MERCURY FROM COPPER.*

By C. ROSCOE SPARE and EDGAR F. SMITH.

At intervals, since the electrolytic separation of mercury from copper was first announced, statements have appeared that the separation was unsatisfactory. The first objection was to the length of time consumed, but it was soon observed in this laboratory that this factor was very materially reduced upon heating the electrolyte to 65° C. Then it was said that the separation was only applicable and satisfactory when the quantity of copper was not too large, as compared with the amount of mercury in the solution. Proper replies were made to these restrictions upon the method, but recently another chemist, in the person of Emil Goecke, published in his inaugural thesis "Ueber den Genauigkeitsgrad elektro - analytischer Arbeitsmethoden, &c.," that he not only required from sixteen to twenty-four hours to effect the separation, but in only three instances did he find the mercury free from copper. It is not the purpose of the writers to question Goecke's ability to conduct electrolytic work, or analytical work of any kind for that matter, but they feel that it is only fair to lay additional actual experience before the reader, leaving time and further trials to determine finally who is right. In this laboratory the method in question has been so frequently carried out with success that it seemed almost useless to repeat; but the following trials were undertaken, and the results are presented in the order in which they were obtained (see Table I.). The details are sufficiently indicated in the tables.

The deposit of mercury was, in each case, tested most carefully for copper, but the latter was never found. Why Goecke encountered the opposite experience we are not prepared to say. The only reagent used for washing was cold distilled water. The source of the electric energy was storage cells. The time factor here, as in numerous other experiments, is much reduced. From the abundance of evidence here presented on this topic and in former communications, we are disposed to leave the controversy to those who delight in such occupation. We submit facts; they alone settle difficulties.

Goecke emphasises the statement that carbon is present in metals deposited by the current from potassium cyanide solutions, "jedoch in so minimalen Mengen, dass dieselben für die Praxis kaum in Betracht kommen." Indeed, in but a single instance, did it attain to 0·1 per cent, so that another writer has said "aber in so minimaler Menge, dass die Versuchsfehler der Bestimmungsmethoden kaum überschritten wurden." And we are disposed to add that in all probability a more thorough washing of the precipitated mercury would have removed the last traces of alkaline cyanide, and the test for carbon would have resulted negatively.

One of the chief purposes of Goecke seems to have been the study of the impurities contained in metals precipitated on the cathode. He points to the occurrence of carbon in the iron deposited from a citrate solution. This fact was commented upon by the person who first precipitated iron from that electrolyte. It is an old story, and before long we shall very probably hear from Dr. E. F.

* Contribution from the John Harrison Laboratory of Chemistry. From the *Journal of the American Chemical Society*, xxiii, No. 8.

TABLE I.

In the approximate proportion of 1 Hg : 1 Cu.

Mercury. Grm.	Copper. Grm.	KCN. Grms.	Dilution. C.c.	Current.	Voltage.	Temperature. °C.	Time. Hours.	Mercury found. Grm.
0·1211	0·1520	2·5	125	N.D.$_{125}$ = 0·03 A	1·2 V / 1·7 V	65°	3½	0·1211
0·1211	0·1520	2·5	125	N.D.$_{125}$ = 0·05 A / 0·03 A *	1·2 V / 1·9 V	60°	2½	0·1208
0·1211	0·1520	2·5	125	N.D.$_{125}$ = 0·03 A / 0·015 A	1·2 V / 1·5 V	63°	3½	0·1211
				1 Hg : 2½ Cu.				
0·1211	0·3040	3·5	125	N.D.$_{125}$ = 0·03 A / 0·015 A	1·2 V / 1·5 V	65°	3½	0·1202
0·1211	0·3040	3·5	125	N.D.$_{125}$ = 0·03 A / 0·02 A	1·2 V / 1·5 V	65°	3½	0·1205
0·1211	0·3010	3·5	125	N.D.$_{125}$ = 0·03 A / 0·02 A	1·2 V / 1·5 V	65°	4	0·1210
				1 Hg : 6½ Cu.				
0·0453	0·3040	3·5	125	N.D.$_{125}$ = 0·03 A / 0·012 A	1·1 V / 1·5 V	65°	2½	0·0453
0·0453	0·3010	3·5	125	N.D.$_{125}$ = 0·03 A / 0·02 A	1·2 V / 1·5 V	65°	3	0·0455
0·0453	0·3040	3·0	125	N.D.$_{125}$ = 0·03 A / 0·01 A	1·1 V / 1·5 V	60°	4½	0·0449
				1 Hg : 11 Cu.				
0·0453	0·5115	6·0	135	N.D.$_{125}$ = 0·03 A / 0·015 A	1·2 V / 1·5 V	65°	2⅝	0·0453
0·0453	0·5115	7·5	135	N.D.$_{125}$ = 0·03 A / 0·015 A	1·1 V / 1·5 V	60°	3½	0·0447
0·0453	0·5115	5·5	135	N.D.$_{125}$ = 0·03 A / 0·01 A	1·2 V / 1·5 V	60°	2½	0·0454
				3 Hg : 1 Cu.				
0·1211	0·0483	2·5	125	N.D.$_{125}$ = 0·022 A / 0·02 A	1·5 V / 1·5 V	60°	2⅝	0·1210
0·1211	0·0483	2·3	125	N.D.$_{125}$ = 0·03 A / 0·01 A	1·5 V / 1·5 V	60°	4⅝	0·1213
0·1211	0·0483	2·0	125	N.D.$_{125}$ = 0·06 A / 0·01 A	1·6 V / 1·5 V	65°	3	0·1205

TABLE II.

Mercury. Grm.	Copper. Grm.	Cadmium. Grm.	KCN. Grms.	Dilution. C.c.	Current.	Voltage.	Temperature. °C.	Time. Hours.	Mercury found. Grm.
0·1211	0·1207	0·1410	4·0	135	N.D.$_{125}$ = 0·04 A / 0·01 A	1·4 V / 1·5 V	65°	3½	0·1204
0·1211	0·1207	0·1410	3·5	135	N.D.$_{125}$ = 0·04 A / 0·01 A	1·2 V / 1·5 V	65°	3½	0·1211
0·1211	0·1207	0·1410	3·0	135	N.D.$_{125}$ = 0·02 A / 0·01 A	1·4 V / 1·5 V	65°	4½	0·1207
0·1211	0·1207	0·1410	3·0	135	N.D.$_{125}$ = 0·03 A / 0·01 A	1·3 V / 1·5 V	60°	4½	0·1203
0·1211	0·1207	0·1410	3·0	135	N.D.$_{125}$ = 0·04 A / 0·01 A	1·4 V / 1·5 V	60°	4	0·1208

TABLE III.

Mercury. Grm.	Copper. Grm.	Cadmium. Grm.	Zinc. Grm.	KCN. Grms.	Dilution. C.c.	Current.	Voltage.	Temperature. °C.	Time. Hours.	Mercury found. Grm.
0·1211	0·1207	0·1410	0·1000	9·0	135	N.D.$_{125}$ = 0·02 A / 0·01 A	1·4 V / 1·5 V	60°	5	0·1206
0·1211	0·1207	0·1410	0·1000	5·0	135	N.D.$_{125}$ = 0·03 A / 0·01 A	1·5 V / 1·5 V	60°	3½	0·1207

* Maximum and minimum currents.

Kern on this subject. He has executed a series of experiments showing upon what this co-precipitation of carbon is due and how it may be avoided. In time, too, we hope to speak of the occurrence of phosphorus in metals deposited from solutions of phosphates. Goecke records it as an ever-present impurity in the metals deposited from such electrolytes.

But to return to the electro-deposition of mercury. The literature relating to this reveals the fact that very little experimentation in this way has been done with mercury in the presence of three or four other metals. Indeed, this is a direction in which investigation could be profitably conducted. We submit results obtained by us in following this thought (see Tables II. and III.).

ANALYSIS OF THE VOLCANIC DUST FROM THE RECENT ERUPTION IN THE WEST INDIES.

THE following is the complete analysis of the sample of volcanic dust which fell over the Barbados on May 8th. It was collected and sent by a correspondent to Mr. Donald F. Mackenzie, a gentleman well known in West Indian circles. The partial analysis appeared in our issue of May 30th last.

SiO_2	51·60
Al_2O_3	21·12
Fe_2O_3	9·28
CaO	9·07
MgO	3·96
Na_2O	0·59
K_2O	0·81
SO_3..	0·89
P_2O_5	0·19
Loss on ignition	1 20
Undetermined	1·29
	100 00

We are also able to give the complete analysis of the dust from the explosion of Mont Pelée, Martinique:—

SiO_2	53·40
Al_2O_3	21·00
Fe_2O_3	9·50
CaO	9·70
MgO	2·00
Na_2O	2·33
K_2O	0 85
SO_3	0·90
P_2O_5	0·25
	99·93

The above analysis was made on a sample dried at 105°, and is of special interest as it was collected from the deck of the S.S. *Roddam* on her arrival at St. Lucia; she was the only ship which escaped from St. Pierre after the eruption.

We regret that we are unable to give the analysis of the dust collected at Roxborough, Grenada, about 90 miles from La Soufrière, St. Vincent; the sample is so small that none can be spared for analysis.

HARD TOOL-STEEL.

By SERGIUS KERN, M.E., St. Petersburg.

IT is well known that not many brands of hard tool-steel can be used with advantage and economy for the preparation of various cutting tools used in the machining of modern armour plates.

I have prepared for such purposes a tool-steel having the following composition:—

	Per cent.
Tungsten	2·00
Molybdenum	0·50
Carbon	0·90
Manganese	0·20
Silicon	0·18

The phosphorus and sulphur must be kept down as low as possible, on the average not more than 0·03 per cent of the combined elements, out of which not more than 0·01 per cent should be sulphur.

The steel was, and must be, prepared by the crucible process.

Such a self-hardening tool-steel is very convenient for the machining of hard metals.

New Admiralty, St. Petersburg, June 6, 1902.

THE RADIO-ACTIVITY OF THORIUM COMPOUNDS.

I. AN INVESTIGATION OF THE RADIO-ACTIVE EMANATION.

By E. RUTHERFORD, M.A., D.Sc.,
Macdonald Professor of Physics, McGill University, Montreal,
and
FREDERICK SODDY, B.A. (Oxon.),
Demonstrator in Chemistry, McGill University, Montreal.

(Continued from p. 272).

The Chemical Aspect of the Question.

THE foregoing furnishes a short review of the physical side of the question at the present time. With regard to the chemical aspect, this has so far not been studied. The photographic method, almost the only one that has until now been used by chemists in the study of radio-activity, is not one which allows of the recognition and differentiation of an emanation as a component factor in producing the phenomena. The photographic method is of a qualitative rather than a quantitative character; its effects are cumulative with time, and as a rule long exposures are necessary when the radio-activity of a feeble agent like thoria is to be demonstrated. In addition, Russell has shown that the darkening of a photographic plate is brought about also by agents of a totally different character from those under consideration, and, moreover, under very general conditions. Sir William Crookes (*Proc. Roy. Soc.*, 1900, lxvi., 409) has sounded a timely note of warning against putting too much confidence in the indications of the photographic method of measuring radio-activity. The uncertainty of an effect produced by cumulative action over long periods of time quite precludes its use for work of anything but a qualitative character.

Two or three chemists have studied the radio-activity of thoria, using the photographic method, without, however, distinguishing between the radio-activity due to the emanation and that due to the thoria itself. Sir William Crookes (*loc. cit.*), who succeeded by an elegant method in separating and isolating the radio-active constituent of uranium, also describes some experiments on thorium compounds with the same object, but did not succeed in effecting a separation. A method based on the fractional precipitation of the sulphate failed completely, but another method, the fractional crystallisation of the nitrate, gave preparations showing a difference in their photographic actions in the ratio of one to three. According to slight variations in the method employed, as, for example, whether a glass or a card bottom was used for the cell containing the substance to be tested (and both seem to have been employed), the radiation from the emanation would or would not contribute largely to the photographic action observed.

Debierne (*Comptes Rendus*, 1900, cxxx., 906), working on a very large scale, obtained from pitchblende, by using reactions which would lead to the separation of thorium, a material different in its chemical properties from radium (barium) and polonium (bismuth), but consisting in great part of thorium. This preparation was 100,000 times more active than uranium, and he therefore assumed the existence of a new element, "actinium," therein. He hazarded the suggestion that the radio-activity of thoria is due to the presence of the same substance, and derived support for this view from the recent work of one of us on the radio-activity of thoria, although on what grounds is not clear.

In the course of their work on the atomic weight of thorium, Brauner (*Trans. Chem. Soc.*, 1898, lxxiii., 951) and Baskerville (*Journ. Am. Chem. Soc.*, 1901, xxiii., 761), have obtained evidence of the presence of a foreign substance associated with thorium. The latter noticed that the separation, as he interpreted it, of this impurity reduced the photographic action considerably, and he concluded

that the pure material would be without photographic action. He employed a modification of Crookes's photographic method, but it cannot be decided with certainty from the description whether the radiation from the emanation would be eliminated or not.

The present work is concerned primarily with the radio-active emanation, although, of course, frequent occasion has arisen to examine correspondingly the ordinary radiation also. The methods employed are of an electrical character, based on the property generally possessed by all radiation of the kind in question, of rendering a gas capable of discharging both positive and negative electricity. These, as will be shown, are capable of great refinement and certainty. An ordinary quadrant electrometer is capable of detecting and measuring a difference of potential of at least 10^{-2} volts. With special instruments, this sensitiveness may be increased a hundredfold. An average value for the capacity of the electrometer and connections is 3×10^{-5} microfarads, and when this is charged up to 10^{-2} volts, a quantity of electricity corresponding to 3×10^{-12} coulombs is stored up. Now in the electrolysis of water 1 grm. of hydrogen carries a charge of 10^{5} coulombs. Assuming, for the sake of example, that the conduction of electricity in gases is analogous to that in liquids, this amount of electricity corresponds to the transport of a mass of 3×10^{-18} grms. of hydrogen; that is, a quantity of the order of 10^{-12} times that detected by the balance. For a more delicate instrument, this amount would produce an inconveniently large effect.

The effects under investigation, from the nature of their manifestation, may well be, and probably are, produced by quantities of matter of the order of magnitude described, and therefore altogether beyond the range of the balance. But to assume on that account that the subject is beyond the pale of profitable chemical investigation is needlessly to limit the field of chemical inquiry. Although surpassing the spectroscope as a detective agent, as a quantitative instrument the electrometer is little inferior in accuracy to the balance. To take as an example the case of thoria mixed with zirconia, the former could be detected and accurately measured by means of its emanation with an electrometer, even although it were only present to the extent of one part in many thousands. A distinction must be made here between *emanation* and *emanating power*. The quantity of the former is what is measured by the electrometer. To express this in terms of weight, the emanating power, that is, the quantity of emanation produced by a given weight of the substance in question, must be known. As will be shown, this value varies with the previous history and present condition of the substance.

The electrometer also affords the means of recognising and differentiating between the emanations of different chemical substances. By the rate of decay, the emanation from thorium, for example, can be instantly distinguished from that produced by radium, and although a difference in the rate of decay does not of itself argue a fundamental difference of nature, the identity of the rate of decay furnishes at least strong presumption of identity of nature.

In the sense that has just been explained, the electrometer can be said to supply the investigation of the property of emanation with methods, so to speak, of quantitative and qualitative analysis which are simple and direct, and there is therefore no reason why the property in question, and even the nature of the emanation itself, should not be the subject of chemical investigation.

Scope of Work.

Of the great number of questions which immediately present themselves for answer in an investigation of this kind, the following are at present claiming our more immediate attention :—

1. Is the power of producing an emanation a specific property of thorium, or is it to be ascribed to the presence of a foreign substance, possibly in minute amount, associated with it and amenable to chemical methods of separation?

2. Can the emanating power of "de-emanated" thoria be regenerated by chemical means? It has been mentioned that thoria, when intensely ignited, loses to a very great extent its power of giving an emanation. If such de-emanated thoria be subjected to a series of chemical changes, will it regain its emanating power or not?

3. Does the emanation or radio-active gas itself possess any property which would associate it chemically with any known kind of gravitational matter?

4. Is it possible to detect, by means of the balance, any loss in weight corresponding to the continuous emission of the emanation or any gain in weight of bodies rendered radio-active thereby?

5. Does the chemistry of thorium present any peculiarity capable of being connected with its almost unique power of producing an emanation?

To interpret rightly the results obtained, a more or less complete study of the effect of chemical and physical conditions on the emanating power is necessary. The effect of the state of aggregation, the presence or absence of water, the influence of light, temperature, the nature of the surrounding atmosphere, the lapse of time since preparation, &c., on the emanating power, as well as the differences in this property exhibited by different compounds, have been investigated.

The present communication does not attempt a full answer to all the above questions. The results so far obtained in answer to the first three will be presented. The work on the fourth is in progress, whilst the results of the investigation of the fifth question will be most conveniently given later in a separate communication.

Electrometer Method of Measuring Emanating Power and Radio-activity.

The term radio-active is now generally applied to a class of substances, like uranium, thorium, radium, and polonium, which have the power of spontaneously giving off radiations capable of passing through thin plates of metal. The radiations are in some cases very complex, but in the case of the substances mentioned, a portion at least of the radiation is similar in all respects to easily absorbed Röntgen rays. The characteristic and general property possessed by these radiations is to produce, from the gas through which they pass, positively and negatively charged carriers, which in an electric field travel to the negative and positive electrodes respectively. In this way, a small current is able to pass through a gas exposed to the radiations, even with a very weak electric field, and the measurement of this current by means of the electrometer affords a means of comparing the intensities of radiation.

As has been mentioned, compounds of thorium (and radium), in addition to radiations which travel in straight lines, emit radio-active emanations, which behave in all respects like a temporarily radio-active gas, and diffuse rapidly through porous substances, as, for example, thick cardboard, which are completely opaque to straight line radiation. Each particle of the emanation behaves as if it were a radiating centre, producing charged carriers throughout the gas in its neighbourhood. The emanation passes through plugs of cotton-wool, and can be bubbled through liquids without appreciable loss of radio-activity, whereas the charged carriers, produced by the emanation in common with the straight line radiation from radio-active substances on the contrary, completely disappear on passing through a plug of cotton- or glass-wool, or by bubbling through liquids. The means of eliminating the effects of the straight line radiation and of measuring the amount of the emanation alone thus suggest themselves. Air passed over uranium or other non-emanating radio-active substance will no longer conduct a current after passage through cotton-wool. The conductivity in the case of thorium, however, will persist, and afford a measure of the amount of emanation present.

Fig. 1 shows the experimental arrangement for com-

paring the emanating power of substances. These are placed in the form of fine powder in a shallow lead vessel inside the glass cylinder, C, 17 c.m. in length and 3·25 c.m. in diameter, provided with indiarubber corks. A current of air from a large gas bag, after passing through a tube containing cotton-wool to remove dust particles, bubbled through sulphuric acid in the vessel, A. It then passed through a bulb containing tightly packed cotton-wool to prevent any spray being carried over. The emanation mixed with air was carried from the vessel, C, through a plug of cotton-wool, D, which completely removed all the charged carriers carried with the emanation. The latter then passed into a long, brass cylinder, 75 c.m. in length and 6 c.m. in diameter. The cylinder insulated on paraffin blocks was connected to one pole of a battery of small lead accumulators, the other pole of which was connected to earth. Three electrodes, E, F, H, of equal length were placed along the axis of the cylinder, supported by brass rods passing through ebonite corks in the side of the cylinder. The current through the gas, due to the presence of the emanation, was measured by means of a Kelvin quadrant electrometer of the White pattern. The electrometer and the connections were suitably screened by means of wire gauze connected to earth. An insulating key was arranged so that either of the electrodes, E, F, H, or all of them together, could be rapidly connected to one pair of quadrants of the electrometer, the other two being always connected to earth.

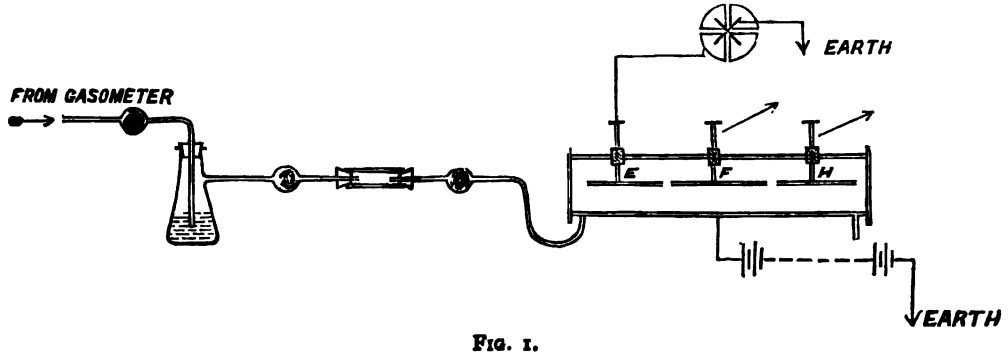

FIG. 1.

The insulation of the electrodes was first tested by sending a current of air through the apparatus without any emanating material in C. The rate of movement of the electrometer needle was accurately observed. On placing the emanating substance in C and continuing the air current for several minutes at a constant rate, the current due to the emanation reached a steady state. On separating the quadrants of the electrometer, the deflection from zero increased uniformly with time. The time taken to pass over 100 divisions of the scale was observed with a stop-watch. The number of divisions passed over per second may be taken as a measure of the current through the gas.

With this apparatus, the emanation from 10 grms. of ordinary thorium oxide produces a current of $3·3 \times 10^{-11}$ ampères between the three electrodes connected together and the cylinder. With the electrometer working at average sensitiveness, this corresponded to a deflection of 100 divisions of the scale in twelve seconds, so that one-hundredth part of this current could be readily measured; that is, the emanation produced by one-tenth of a grm. of thorium oxide.

An electrometer one hundred times more sensitive than this failed to detect the presence of an emanation or radio-activity in the oxides of tin, zirconium, and titanium, the other elements of the same group in the periodic table.

Variation of the Current with Voltage.—The current through the gas observed with the electrometer at first increases with the voltage, but a stage is soon reached when there is a very small increase for a large additional voltage. This is one of the most characteristic properties of conducting gases. For small voltages, only a small proportion of the charged carriers reach the electrode, on account of their re-combination throughout the volume of the gas. When the electric field is increased until all the carriers reach the electrode before any appreciable re-combination can occur, the current is at a maximum, and remains constant for large increases of voltage, provided, of course, that the electric field is below the value necessary for a spark to pass. In the experimental case, a pressure of 50 volts was found sufficient to give the maximum current between the electrodes.

This property of conducting gases allows us at once to make sure that the insulation of the electrodes is perfect at all stages of a long experiment; 100 volts applied instead of 50 to the cylinder should give the same current if the insulation is unaffected.

Rate of Decay of the Radiation from the Emanation.—The three electrodes, E, F, H, were used to compare the "rates of decay" of the radiations from the emanations of different substances. In the previous papers quoted, it has been shown that the radiating power of the thoria emanation falls to half its value in about a minute. In consequence of this, the current observed for the electrode E is greater than for electrode H. Knowing the velocity of the current of air along the cylinder and the respective currents to the electrodes E, F, H, the rate of decay of the radiation can be readily deduced. If, however, we merely require to compare the rate of decay of one emanation with another, it is only necessary to compare the ratio of the currents to the electrodes E, F, H in each case, keeping the current of air constant. If the ratio of the currents is the same we may conclude that the radiating power of each diminishes at the same rate. The comparison of emanation is thus rendered qualitative as well as quantitative. In most of the experiments, the current to the electrode E was about twice that to the electrode H; the velocity of the current of air along the cylinder was thus about 0·8 c.m. a second.

Comparison of Emanating Power.—The experiments in all cases on the amount of emanation from different substances are comparative. The standard of comparison was usually a sample of 10 grms. of thoria as obtained from the maker, which gave out a conveniently measurable quantity of emanation. Preliminary experiments were made to find the connection between the weight of thoria and the amount of emanation as tested in the cylinder. The following numbers show that the amount of emanation is directly proportional to the weight of the substance:—

Weight of thoria. Grms.	Divisions of scale per second.
2	1·41
4	2·43
10	6·33
20	13·2

This result shows that within the limit of accuracy desired we may take the amount of emanation as directly proportional to the weight of the substance. The determinations in the above table were made with the three electrodes connected together with the electrometer, and with a constant flow of air. The lead vessel in which the thoria was placed was 7·4 by 3·5 c.m. in area and 3 m.m. deep. In the comparison of emanating power, the maximum current between the electrodes for the standard 10 grms. of thoria was first observed. This was removed, and a known weight of the specimen to be tested was substituted, and the deflections again observed after the conditions had become steady.

If d_1 = No. of divisions per sec. for a weight, w_1, of thoria;
d_2 = No. of divisions per sec. for a weight, w_2, of the specimen;
then—

$$\frac{\text{Emanating power of specimen}}{\text{Emanating power of thoria}} = \frac{d_2 w_1}{d_1 w_2}.$$

The values d_1 and d_2 are corrected, when necessary, for natural leakage; that is, the current which passes under similar circumstances when no emanating material is present. This current is chiefly made up of a leakage due to conduction over the ebonite, as well as the current produced by the excited radio-activity which has collected on the negative electrode during the course of the day's experiments. It is generally very small, and the correction is only necessary when a specimen of substance almost free of emanation is being tested.

An example taken at random from the note-book will serve to illustrate the method of calculating the results, the emanating power of the comparison sample being considered 100 per cent:—

Dec. 7th, 11 *a.m.*—Natural leakage	10 divisions in	50"	
	0·20	,,	1"
5 grms. comparison sample ThO_2	100	,,	23·5"
3·6 ,, ThO_2 ignited twenty-four hours over Bunsen burner in platinum crucible	50	,,	35·2"
d_1 = 4·25, corrected for nat. leakage = ...			4·05
d_2 = 1·42 = ...			1·22

$$\frac{d_2 w_1}{d_1 w_2} = 0{\cdot}42, \text{ or } 42 \text{ per cent.}$$

(To be continued).

CONTRIBUTIONS TO THE CHEMISTRY OF THE RARE EARTHS OF THE YTTRIUM GROUP.*

I.

By L. M. DENNIS and BENTON DALES.

(Continued from p. 266).

Experimental.

In 1877 J. W. Mallet published an article on sipylite, a new niobate, from Amherst County, Virginia (*Am. Journ. Sci.*, 1877, x v., 397). An analysis of the mineral made under his direction by W. G. Brown indicated the substance to be essentially a niobate of erbium containing some tantalic acid, about 1 per cent of yttrium oxide, and approximately 11·5 per cent of the earths of the cerium group.

In the following year Delafontaine (*Comptes Rendus*, lxxxvii., 933) examined this mineral and stated that it contained erbium, philippium, and ytterbium, the last-named element having been isolated but a short time before by Marignac.

A supply of sipylite which had been procured by one of us from Virginia and Texas was used as the source of the earths in this investigation. The mineral was finely powdered and was then mixed with a large excess of primary potassium sulphate, and this mixture was fused in deep iron dishes in a crucible furnace. When decomposition was complete, the mass was cooled, powdered, and thrown into ice-water, the water being stirred until complete solution of all soluble compounds had been effected. The clear supernatant liquid was then syphoned off, nearly neutralised with ammonia, and precipitated with oxalic acid. The oxalates were then washed, dried, ignited, dissolved in sulphuric acid, and precipitated with ammonium hydroxide. This precipitate, after thorough washing by decantation, was dissolved in nitric acid, and to the neutral solution there was then added a saturated solution of potassium sulphate and crystals of the same salt. The mixture was violently stirred until the supernatant liquid failed to show the absorption bands of didymium. The resulting precipitate of the earths of the cerium group was removed by filtration, and was washed with a saturated solution of potassium sulphate. The filtrate was diluted with water and was precipitated by a solution of oxalic acid. These mixed oxalates, which, of course, still contained traces of the earths of the cerium group, were dried and ignited. The resulting mixture of oxides weighed about 350 grms. and was of a deep orange-yellow colour.

The atomic weight of the oxide mixture, assuming that all the oxides present were of the R_2O_3 type, was determined by the oxalate method of Gibbs and the sulphate method of Krüss, and was found to be in the neighbourhood of $R^{III} = 108$. Assuming that the average atomic weight of the elements other than yttrium lies in the neighbourhood of 165, this result shows that the mixture consists largely of yttrium oxide, approximately 75 per cent. The absorption spectrum when observed through a 10 c.m. layer of the saturated solution of the nitrate of the mixture showed the bands tabulated. The measurements in the table coincide with the maximum intensities of the absorption bands unless otherwise indicated.

[A description of the grating spectroscope and method of measuring the absorption bands, is here omitted].

The Determination of the Atomic Weights of the Elements.

The method described by Gibbs (*Am. Chem. Journ.*, xv., 547) was used when only an approximate result was desired. The procedure was as follows:—A neutral or only very slightly acid solution of the earths whose atomic weight is to be determined is diluted with water, brought nearly to the boiling-point, and is then precipitated with a hot dilute solution of pure oxalic acid. The precipitate is washed by decantation and on the filter with hot water to remove the excess of oxalic acid, and is then dried at a temperature of 125°. A weighed portion of the oxalate precipitate is then converted to the oxide by ignition, and this oxide is weighed. Another weighed portion of the oxalate is dissolved in dilute sulphuric acid (1:8) and the oxalic acid is determined by titration with potassium permanganate. The atomic weight of the earths in the mixture of the rare earths is then easily calculated from the ratio $R_2O_3 : C_2O_3$. This method of determining the atomic weights gives results that are sufficiently accurate to enable one to judge of the progress of a method of separation or fractionation, but which are far from being exact; the comparison of the method with the sulphate method showed that the Gibbs procedure gave results differing by as much as four units from the sulphate method of Krüss. To ascertain the effect of changes in the conditions prevailing at the time of precipitation the following experiments were made:—

A solution of the earths was carefully freed from other elements by precipitating it with hydrogen sulphide, filtering off a very slight precipitate which formed, boiling the filtrate to remove the hydrogen sulphide, and precipitating it with ammonia, dissolving the washed precipitate in hydrochloric acid, and after nearly neutralising with ammonia precipitating this solution with oxalic acid. The

* *Journal of the American Chemical Society*, vol. xxiv., No. 5.

Angle.	Wave-length λ.	Element.	Description of band.
12° 2′ 30″	6832·4	{Erbium Thulium	Faint, rather broad, edges vague.
11° 45′ 10″	6670·7	Erbium	Slightly stronger than 6832·4.
11° 29′ 00″	6519·9	Erbium	Moderately broad and strong, with two maxima; one in centre, the other at right edge.
11° 25′ 20″	6485·7		
11° 16′ 40″	6404·7	Holmium	Moderately strong; right edge sharp, maximum to right of centre.
10° 11′ 50″	5797·9	Didymium	Edges of broad, very hazy band. Scarcely visible.
10° 4′ 00″	5725·5		
9° 39′ 50″	5497·7	Unknown (Crookes)	Faint.
9° 30′ 30″	5410·0	Erbium	Broad strong band; two maxima, one to left of centre, the other at right edge.
9° 25′ 30″	5363·0	Holmium	
9° 11′ 10″	5228·2	Erbium	Strongest band in spectrum; two maxima, one at left edge, the other in centre. Right edge indistinct.
9° 6′ 40″	5185·9		
8° 32′ 20″	4862·7	Samarium	Almost as strong as 5228·2. Extends from λ 4938·1 to λ 4825. Maximum to left of centre.
8° 17′ 10″	4719 8	Samarium	Very faint.
8° 11′ 50″	4669·5	Samarium	Very faint.
7° 54′ 00″	4501·3	Dysprosium	Almost as strong as 5228·2. Extends from λ 4550·0 to λ 4449·4.
7° 43′ 40″	4403·8	Didymium (?)	Very faint.
7° 16′ 50″	4150·3	Samarium	Faint and hazy.

oxalates thus obtained were washed with very dilute hydrochloric acid (0·1 per cent) to remove iron, then with water, and were then dried, ignited, and the resulting oxides were dissolved in hydrochloric acid. This chloride solution was divided into two parts. One was precipitated with a hot and dilute solution of oxalic acid, while the other was precipitated in the cold and in more concentrated condition with a cold concentrated solution of oxalic acid. Another portion of the same original material was purified and treated in exactly the manner described above, and was divided and precipitated in the same way. The results of the atomic weight determinations of these various portions by the oxalate method were as follows:—

	Hot.	Cold.
1	111·28	108·26
2	105·06	104·70

The variations in these results show that they are greatly influenced by changes in the conditions, and that they by no means agree even when the same conditions pertain.

The other method employed for the determination of the atomic weights was that described by Krüss (*Zeit. Anorg. Chem.*, iii., 46). In this procedure the oxalate of the earth is placed in a porcelain crucible and ignited over a blast-lamp to constant weight. The crucible containing the oxide is placed on a water-bath, covered with a funnel, and the heating of the water-bath is continued until the water vapour has slowly and completely slaked the oxide. The substance is then dissolved in the crucible with dilute hydrochloric acid. A moderate excess of dilute sulphuric acid is added, and the solution is concentrated as far as possible on the water-bath. The excess of sulphuric acid is removed by placing the crucible on an iron plate 5 m.m. thick and heating the plate with a small Bunsen flame. This is continued until constant weight is obtained, and from the ratio of the oxide to the anhydrous sulphate, the atomic mass of the earth or earths present is calculated. Considerable difficulty was experienced in making duplicate analyses agree satisfactorily. The probable explanation of this trouble is to be found in the article by Brauner and Pavlicek (*Proc. Chem. Soc.*, xvii., No. 235, p. 63), in which those authors call attention to the fact that the sulphate which is finally weighed is liable to contain some acid sulphate. This acid sulphate is so stable that some of it remains undecomposed even at a temperature above 500°, while in other parts of the crucible it may have been partially broken down into a basic sulphate. They state that the error is greatest in the case of lanthanum, and that it decreases as the basicity of the earth decreases. They determine the amount of sulphuric acid which is present in excess over the amount necessary to form the normal sulphate by dissolving the dry sulphates in water and titrating the free sulphuric acid with a twentieth normal solution of sodium hydroxide, using ethyl-orange as an indicator. The normal sulphate obtained by repeated crystallisation was found by them to be neutral to ethyl-orange.

These statements concerning the sulphates of the earths of the cerium group hold true for those of the yttrium group, but the error is less because the basicity of the earths of this group is less. For example, two weighed portions of a sulphate which had given R^{III} = 115·90 and 116·42, were dissolved in water and the excess of free acid titrated as above, using methyl-orange as an indicator. The corrected results for these two samples were R^{III} = 116·19 and 116·47. Another set which had given R^{III} = 141·22 and 141·53 gave after correction 141·51 and 141·73. A third set giving R^{III} = 107·31 and 106·75 gave after correction 107·37 and 106·92. There seems to be no regularity in the amounts of acid sulphate which are formed. The maximum error caused by its presence seems to be about 0·3 of a unit. All of the solutions which were tested showed an acid reaction, but in some cases a drop or two of the solution of sodium hydroxide was sufficient to neutralise the free acid. The results of duplicate determinations were usually brought to closer agreement by the introduction of the correction. In only one case was the opposite effect noted.

Experiments upon Methods for Separating Yttrium Group Earths.

The aim which the authors had in view in this work was not so much the isolation of one or another constituent of the mixture of the rare earths, but rather the study of various methods of separation to ascertain along what lines different treatments will cause the earths to separate. A complete systematic study of the different methods of separation was also not undertaken, for those procedures which give slow results and in which the successive treatments are of but slight effect upon the rare earth mixture, cannot be successfully employed, because of the time which is consumed and of the incompleteness of the separation which they bring about. Therefore, in the trials of the different methods to be described below it will be seen that the procedure was abandoned as soon as it became apparent that only slow separation was being effected.

(To be continued).

NOTICES OF BOOKS.

Les Nouveautés Chimiques pour 1902. Par C. POULENC, Docteur ès sciences. One vol., 8vo, 344 pp., 214 figures. Paris: J. B. Baillière and Son.

In this volume we have, methodically arranged, new laboratory apparatus and methods applicable to scientific and industrial research. In the first chapter are collected together new appliances in physical chemistry, such as apparatus for determinations of density, high temperatures, fusion-points, &c.

The second chapter comprises apparatus for chemical manipulations, such as gas-burners, drying stoves, extraction apparatus, and appliances for the production of gases.

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Finally, in the fifth chapter are collected together all appliances useful to bacteriology.

Chemists cannot fail to find in this volume much that will be useful to them in everyday work. There is a good table of contents and an exceptionally good index.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxiv., No. 20, May 20, 1902.

Hydrogenation of Ethylenic Carbides by the Method of Contact.—Paul Sabatier and J. B. Senderens.—It is apparent that the direct hydrogenation can only be effected by means of copper for the α-ethylenic carbides; that is to say, for those in which one of the CH_2 groups remains non-substituted. Styrolene or phenylethylene, $C_6H_5.CH=CH_2$, belongs to this class, and the authors recently showed that copper effects the transformation of this substance regularly into ethylbenzene. Limonene is generally supposed to possess the formula—

$$CH_3—C_6H_8—C\begin{matrix}CH_3\\ \leqslant CH_2\end{matrix}.$$

From this it can be predicted that copper cannot effect the hydrogenation of the cyclic radicle, but that it acts on the α-ethylenic branch. Such is found to be the case. Prolonged action of hydrogen towards 190° in presence of copper changes limonene into a carbide, $C_{10}H_{18}$, which boils at 170° and whose density d^0_0 is 0·827. This carbide is violently attacked by nitrosulphuric acid. Hydrogenation in presence of nickel transforms it into methyl-paraisopropyl (cyclohexane); this latter being an isomer of the position of methane.

Action of Sulphites on Nitroprussiates. Bœdeker's Reaction.—Juan Fages.—To recognise the presence of sulphites the red colouration which they communicate to a solution of sulphate of zinc containing a little sodium nitroprussiate is employed. The addition of a little potassium ferrocyanide increases the sensitiveness of this reaction of Bœdeker. It is known that the property causing the formation of the red body belongs exclusively to the chemically neutral sulphite. Acid sulphites completely prevent the neutral sulphite from forming it. Consequently, to investigate a sulphite, the solution, if acid, must be neutralised, preferably with a neutral carbonate; free alkalis retarding the reaction. The author undertakes a careful examination of such reactions,

Volumetric Estimation of Iodides in presence of Chlorides and Bromides.—V. Thomas.—The author devises a new method of volumetric estimation of iodides in presence of chlorides and bromides by operating in presence of excess of a thallic salt. The iodine set free is eliminated by merely boiling the liquid. When all the iodine is got rid of, the non-reduced thallic salt is titrated. If a known quantity of thallic salt is used to start with and the difference expressing the quantity of $TlCl_3$ transformed into TlCl, the amount of iodide originally in the liquid can easily be deduced. This method of estimation of iodides is very exact and very rapid if a dilute solution of a thallic salt is employed.

A Gradual Method of Synthesis of Aldehydes.—L. Bouveault and A. Wahl.—By treating α-ethyl-nitrodimethylacrylate with sodium in presence of damp ether, or better with caustic alkalis at 50—60°, the authors succeeded in decomposing this substance into carbonic acid, alcohol, and a nitroisobutylene—

$$\begin{matrix}CH_3\\ CH_3\end{matrix}\!\!>C=CH—NO_2.$$

This synthesis has the advantage of determining the constitution of nitroisobutylene which Haitinger's preparation leaves doubtful. The authors intend to generalise this synthesis.

MISCELLANEOUS.

Science Abstracts.—The following Journals have been added since the beginning of 1902 to the list regularly dealt with in *Science Abstracts*, making a total at present of about 150:—

Journal of the Russian Physico-Chemical Society (Russian).
Proceedings of Academy of Sciences, Amsterdam (Dutch).
" " " Stockholm (Swedish).
" " " Christiana (Norwegian).
" " " Copenhagen (Danish).
Also—
Zeitschrift für Anorganischen Chemie.
Royal Society of Edinburgh Transactions and Proceedings.
British Optical Journal.
Elettricista (Rome).
Iron and Steel Institute Transactions.
American Society of Mechanical Engineers Transactions.
Engineering and Mining Journal (New York).
Power (New York).
American Telephone Journal.
La France Automobile.
Der Moterwagen.
Automobile Review (Chicago).
Archives of the Röntgen Ray.
Philosophical Society of Washington Bulletin.

The Solubility of Salts; the Iodates of Cobalt and Nickel.—A. Meusser.—Having taken up the study of these iodates the author finds that that of cobalt IO_3Co only exists in three forms:—(1) The blue-violet crystalline anhydride, forming in warm, very acid solutions; (2) that with $2H_2O$, in red-violet crystalline crusts; and (3) that with $4H_2O$, in crusts formed of hexagonal prisms. These two latter hydrates have solubilities increasing with the temperature, which is contrary to that of the anhydrous salt which decreases with the temperature. The anhydrous salt once formed only shows a feeble tendency to become hydrated. Facts very similar to these are observed with the nickel salt, IO_3Ni, which is also obtained in the anhydrous form in small, pale-yellow needles; then with $2H_2O$, but under different forms, one in green spherocrystalline crusts forming at 25—30°, the other at 50°, in small brilliant prisms; finally, with $4H_2O$ at 0—10° in hexagonal prisms. The curves of solubility have the same direction as those of the corresponding salts of cobalt.—*Berichte*, vol. xxxiv., p. 2432.

The Solubility of Salts. The Chromates of Calcium.—F. Mylius and J. von Wrochem.—A solution of CrO_4Ca in an excess of CrO_3, evaporated in a desiccator, gives a tetrachromate, $CaO.4CrO_3+6H_2O$, in the form of very soluble, deep red crystals. The dichromate, Cr_2O_7Ca, crystallises with 3 H_2O, and also with 4 H_2O; this latter hydrate is a new one, and occurs in beautiful orange-coloured prisms. At the other end of the series there appears a basic chromate, $2CaO.CrO_3$, already described by M. von Foullon; but the most important one is the neutral chromate CrO_4Ca, which can exist in several different degrees of hydration, of which the following are the solubilities at 18°:—

	Density of the saturated solutions.	Molecules of the salt per 100 mols. of water.
Hydrate with 2 H_2O (α-clinorhombic variety .	1·149	1·93
Hydrate with 2 H_2O (β-orthorhombic variety.	1·105	1·33
Hydrate with H_2O ..	1·096	1·22
Hydrate with $\frac{1}{2}$ H_2O ..	1·044	0·51
The anhydrous salt ..	1·023	0·27

The hydrate $2H_2O$, variety *a*, is in very small crystals, macled like gypsum, with which it is isomorphous. It is very unstable, and has a tendency to pass to the β form or to one of the lower hydrates spontaneously. The hydrate $\frac{1}{2}H_2O$ corresponds to a product of heating gypsum, and is obtained by rapidly heating the 15 per cent solution of CrO_4Ca, treated with $CaCl_2$ or with glycerin, to 100°. It has the appearance of chloroplatinate of ammonium; it does not lose its water below 400°. The solubility of the different hydrates decreases fairly rapidly with the temperature.—*Berichte*, vol. xxxiii., p. 3689.

NOTES AND QUERIES.

*** Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

River Water.—If Mr. Ernest A. Lewis cares to communicate with me I can give him the potable analysis of two samples of South and West Jaquel I did in 1897.—Leo Taylor, F.I.C., 31, Moorgate Street, London, E.C.

Extraction of Oil from Seeds.—Can any reader recommend a good book dealing mainly with the extraction of oil from seeds by hydraulic pressure? I have Thorpe's "Dictionary," Grove and Thorpe's "Technology," Alder Wright's "Soap." and Hurst's "Oils, &c.," but they do not give sufficient detail.—Seed Crushing.

MEETINGS FOR THE WEEK.

Wednesday, 18th.—Chemical, 5.30. Ballot for the Election of Fellows. "Elimination of a Nitro-group on Diazotisation—Dinitro-*p*-anisidine," by R. Meldola and J. V. Eyre. "A New Type of Substituted Nitrogen Chlorides," by F. D. Chattaway. "The Colour Changes exhibited by the Chlorides of Cobalt and some other Metals, from the Standpoint of the Theory of Electro-affinity," by F. G. Donnan and H. Bassett, jun. "An Accurate Method of Determining the Compressibility of Vapours," by B. D. Steele. "The Molecular Condition of Borax in Solution," by H. S. Shelton. "Preliminary Notice of some New Derivatives of Pinene and other Terpenes," by W. A. Tilden and H. Burrows. "The Preparation of Pure Chlorine and its Behaviour towards Hydrogen," by J. W. Mellor and E. J. Russell.

— Microscopical, 8. "The Genus Synchæta," by C. F. Rousselet.

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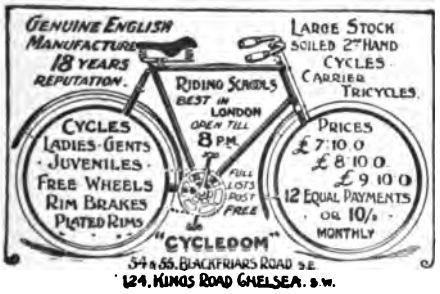
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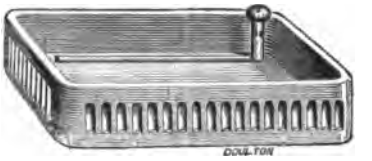

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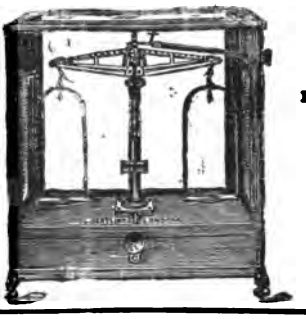

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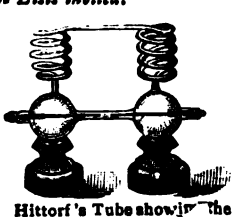

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THE CHEMICAL NEWS.

Vol. LXXXV., No. 2221.

COEFFICIENTS OF THE CUBICAL EXPANSION OF ICE, HYDRATED SALTS, SOLID CARBONIC ACID, AND OTHER SUBSTANCES AT LOW TEMPERATURES.*

By JAMES DEWAR, M.A., D.Sc., LL.D., Jacksonian Professor, Cambridge University, and Fullerian, Royal Institution, London.

(Concluded from p. 279).

Notes on the Results of Table I.

Sodium, extending down to low temperatures, has a coefficient about the same as that of mercury at the ordinary temperature. The coefficient for sulphur is about half of that between 0° and 100°, being 0·0002237, and that of iodine is not far removed from the value 0·000285 given for the solid at ordinary temperatures. The rate of expansion of liquid iodine is about three times this value. Paraffin ought to have a value of 0·0004633 from Fizeau, but Rodwell's coefficient between 0° and 38° is 0·00035. The value found for naphthalin is about half that of the liquid near its melting-point, viz., 0·000785. If the liquid coefficient be taken at a corresponding temperature to that of the liquid carbonic acid when comparing it with the solid, then its value is 0·001213, or the coefficient would be now in the ratio of 4 to 1. The graphite calculated from Fizeau should be 0·0000929, which is greater than my value; but the samples were different. My two specimens of chloride of ammonium gave nearly the same value, and the result is in agreement with that found by Playfair and Joule, viz., 0·000191. If a Fizeau coefficient for this salt is calculated, the value is 0·0000761, which in this case is far too small. The coefficient found for oxalic acid is again only a little smaller than that given by Playfair and Joule, viz., 0·0002748. As regards the hydrated salts, phosphate of soda, hyposulphate of soda, and

* A Paper read before the Royal Society, May 1, 1902.

Table I.

	A–C.	A–B.	D.	d.†	$d_{17°}$.	a 0·000
Sulphate of aluminium (18)*	45·338	26·961	1·0225	1·7194	1·6913	0811
Biborate of soda (10)	47·057	28·328	1·0405	1·7284	1·6937	1000
Chloride of calcium (6)	68·942	37·493	0·9347	1·7187	1·6775	1191
Chloride of magnesium (6)	36·663	22·438	0·9816	1·6039	1·5693	1072
Potash alum (24)	50·462	30·177	0·9816	1·6414	1·6144	0813
Chrome alum (24), large crystal ..	47·345	26·406	1·0226	1·8335	1·8199	0365
Chrome alum (24)	40·147	23·665	1·0517	1·7842	1·7669	0478
Carbonate of soda (10)	55·675	34·863	0·9347	1·4926	1·4460	1563
Phosphate of soda (12)	57·085	35·848	0·9700	1·5446	1·5200	0787
Hyposulphate of soda (5)	53·121	28·836	0·9573	1·7635	1·7290	0969
Ferrocyanide of potassium (3) ..	55·914	29·115	0·9887	1·8988	1·8533	1195
Ferricyanide of potassium	68·902	35·961	0·9887	1·8944	1·8109	2244
Nitro-prusside of sodium (4)	49·614	28·526	0·9887	1·7196	1·6803	1138
Chloride of ammonium, sample i...	34·530	21·032	0·9597	1·5757	1·5188	1820
Chloride of ammonium, sample ii..	31·083	18·870	0·9597	1·5809	1·5216	1893
Oxalic acid (2)	23·644	13·293	0·9571	1·7024	1·6145	2643
Oxalate of methyl	56·465	37·760	1·0217	1·5278	1·4260	3482
Paraffin	15·675	16·031	0·9992	0·9770	0·9103	3567
Naphthalin	43·622	33·001	0·9347	1·2355	1·1589	3200
Chloral hydrate..	53·321	26·436	0·9789	1·9744	1·9151	1482
Urea	42·811	32·326	1·0282	1·3617	1·3190	1579
Iodoform	140·012	32·381	1·0282	4·4459	4·1955	2930
Iodine..	17·806	4·135	1·137	4·8943	4·6631	2510
Sulphur	25·499	13·813	1·137	2·0989	2·0522	1152
Mercury	77·719	5·135	0·9502	14·382	14·193‡	0887
Sodium	16·904	16·504	0·9818	1·0066	0·972	1865
Graphite (Cumberland)	22·221	9·986	0·9573	2·1302	2·0990	0733

* The figures ( ) refer to the number of molecules of water of crystallisation.
† Density at the temperature of liquid air.
‡ At −38·85°.

Table II.—*Ice.*

Silver ball. Weight lost in— 1. Oxygen; 2. Liquid air.	Density of liquid air.	Weight of ice and copper ball and wire. Gaseous air at −188·7° C.	Weight of ice and copper ball and wire. Liquid air at −188·7° C.	Weight of copper ball and wire. Air at 16° C.	Weight of copper ball and wire. Liquid air −188·7° C.	Weight of ice *in vacuo*.	Density of ice at −188·7° C.
1. 14·411	0·9820	47·080	33·430	38·075	33·933	9·0612	0·93032
2. 12·446							
1. 14·409	0·9502	46·78	33·88	38·128	34·103	8·706	0·92646
2. 12·042							
1. 14·407	0·9942	56·391	32·740	38·127	33·94	18·167	0·93318
2. 12·597							

Table III.—*Solid Carbonic Acid.*

Weight of solid CO_2. Gaseous air at −188·8° C.	Weight of solid CO_2. Liquid air at −188·8° C.	Density of liquid air.	Weight *in vacuo*.	Density at −188·8° C.
19·865	7·905	0·9832	19·919	1·6308
22·55	9·37	0·9502	22·61	1·6226

chloride of calcium, having the respective values 0·0001384, 0·0001516, and 0 0006887, as found by Kopp, the low temperature coefficients are much smaller in each case. With the exception of carbonate of soda and chrome-alum, all the other hydrated salts have a coefficient of expansion not differing greatly from that of ice at low temperatures. Generally, the densities of the compressed blocks of different bodies agree well with the results of other observers, but my potash-alum had only a density of 1·614, whereas Playfair and Joule give 1·731. It will be noted that iodoform is a highly expansive body like iodine, and that oxalate of methyl has nearly as great a coefficient as paraffin, which is one of the most expansive solids. The correcting factor was used for paraffin, naphthalin, chloral hydrate, iodoform, and sodium. All weights are in grms.

It will be possible by cooling the moulds with liquid air during the process of hydraulic compression, to produce cylindrical blocks of solid bodies of lower melting-points than any given in this investigation, such as alcohol, ether, nitrous oxide, ammonia, chlorine, &c., and to ascertain their coefficients of expansion in the solid state between the individual melting-points and the boiling-point of liquid air.

This method, which works well with liquid oxygen or air, fails when applied to liquid hydrogen, as the density of the liquid is too small (apart from other difficulties) to give accurate values of the weights of fluid displaced. For temperatures about 20° absolute, recourse must be had to measurements of the coefficient of linear expansion, and such observations could only be applied with ease to metallic bodies and alloys.

THE PRESENCE OF TELLURIUM IN INGOTS OF AMERICAN SILVER.

By CAMILLE VINCENT.

It has so happened that I have had to determine the cause of the bad quality of some silver ingots of high standard coming from America.

The metal was very brittle, and cracked on filing. The silver was fused with 5 per cent of nitrate of potassium in order to oxidise the impurities, and after being treated twice in this manner it assumed a normal malleability.

It occurred to me that the bad quality of this silver might be due to the presence of a small quantity of selenium in the same manner as previously observed by Debray on certain similar silver ingots. Therefore, I examined the slag resulting from the fusion of the metal with saltpetre with a view to testing for selenium.

The material was exhausted with boiling water, and the filtrate, acidulated with hydrochloric acid, was evaporated to dryness to make insoluble the precipitated silica, due to the attack of the sides of the crucible by the saltpetre.

The mass, taken up with water, gave a solution which, after filtration, was treated with bisulphite of sodium and hydrochloric acid; this treatment produced a greyish precipitate which increased for several hours.

This precipitate, washed by decantation and then dried, was recognised as being pure tellurium free from all trace of selenium; it gave the violet colouration with hot, concentrated sulphuric acid, which, as is well known, is characteristic of tellurium.

When roasted in an open tube the product gave tellurous anhydride, which is easily recognised.

The ingots examined came from a mineral containing hessite (telluride of silver), and the metallurgical treatment followed, not having been sufficiently perfect, had left a small quantity of tellurium in the silver, and this had seriously modified its physical properties.—*Bull. Soc. Chim.*, Series 3, vol. xxvii., No. 2.

SOME ANALYSES OF COMMON PIGMENTS.

By EDWY G. CLAYTON, F.I.C., F.C.S.

The following analyses show how considerable are the variations which may be met with in such ordinary pigments as zinc-white, Venetian-red, and the like.

No. 1 was crude zinc-white, yellowish in colour. No. 2, though of poor quality and grey tinted, was somewhat better than No. 1. No. 3 was altogether a more satisfactory product; but, like Nos. 1 and 2, it contained a notable proportion of lead oxide. A little metallic lead also was present in 1 and 2. The constitution of No. 4 differed altogether from that of the other samples; zinc sulphide taking the place of zinc oxide, while a large proportion of insoluble matter was present. No doubt there would be great differences in price between the several samples, and the pigments were sold, not under the name of zinc oxide, but as "zinc-white," &c. The possibility of the presence of appreciable quantities of lead or lead oxide in some samples of a pigment which is often chosen because of its supposed freedom from that metal, should be borne in mind; and, in cases of importance, tests should be made.

One of the specimens of Venetian-red contained about 50 per cent of gypsum, and the other much silica. The burnt umber was probably not the natural umber calcined, which would have contained more manganese and less iron oxide, but a manufactured product.

	Crude zinc-white. (1).	Crude zinc-white. (2).	Zinc-white. (3).	Zinc-white. (4).	Venetian-red. (a).	Venetian-red. (b).	Burnt umber.
Hygroscopic water	10·20	0·23	0·21	8·95	1·61	9·83	3·70
Combined water	12·62	0·14	0·49		1·78	4·36	6·24
Zinc oxide	69·68	92·43	95·98	—	—	—	—
Zinc sulphide	—	—	—	30·48	—	—	—
Metallic zinc	trace	2·76	—	—	—	—	—
Metallic lead	0·08	0·47	—	—	—	—	—
Lead oxide (litharge)	6·57	3·97	3·14	—	—	—	—
Insoluble matter	0·77	—	—	59·89	64·28	14·70	7·91
Alumina	0·08	—	—	0·68	—	3·73	—
Oxide of iron	trace	—	—	trace	27·01	19·28	79·52
Lime	—	—	—	—	—	—	0·37
Manganese dioxide	—	—	—	—	—	—	2·26
Magnesia	—	—	—	—	1·32	0·68	—
Calcium sulphate	—	—	—	—	3·03	43·90	—
Calcium carbonate	—	—	—	—	0·97	3·52	—
Carbon dioxide	—	trace	0·18	—	—	—	—
	100·00	100·00	100·00	100·00	100·00	100·00	100·00

CONTRIBUTIONS TO THE CHEMISTRY OF THE RARE EARTHS OF THE YTTRIUM GROUP.*

I.

By L. M. DENNIS and BENTON DALES.

(Continued from p. 286).

Fusion of the Yttrium Group Nitrates with Alkali Nitrates.

THE method first tried was that of fusion of the rare earth nitrates with alkali nitrates, proposed by Debray, and which, as modified by Dennis and Magee (*Journ. Amer. Chem. Soc.*, xvi., 653), gives such satisfactory results in separating cerium from the other members of the group. It was hoped that the decomposition points of some of the yttria group double nitrates might be found sufficiently far apart to admit of a sharp separation, but such was not the case.

The dry yttrium group nitrates were mixed with six times their weight of a molecular mixture of potassium and sodium nitrates, placed in a deep porcelain evaporator, and fused in a large double air-bath, the space between the walls of the bath being filled with infusorial earth to prevent radiation. Three fusions were made, and after each the substance was extracted with hot water, yielding an insoluble and a soluble portion. Fusions were made at three temperatures, 280°, 320°, and 360°, using different mixtures each time. These temperatures were measured by means of a Le Chatelier pyrometer manufactured by Keiser and Schmidt, of Berlin. Each portion was fused for about three hours. The results were irregular, and gave no indication of a sharp separation by this method, as the following atomic weights show:—

Atomic weight of the initial metal mixture, 108·19.

Insoluble.	Temperature.	Soluble.
114·70	280°	108·31
127·55	320°	108·14
112·24	360°	102·43

A comparison of the absorption spectra of solutions of these portions and of the original material with a direct-vision spectroscope showed no appreciable differences.

Partial Precipitation with Hydrochloric Acid Gas.

Partial precipitation of the anhydrous chlorides by hydrochloric acid gas from a cooled solution was next tried. A concentrated solution of the chlorides (R^{III} = 107·94) was placed in a gas wash-bottle surrounded by a freezing-mixture of ice and salt; hydrochloric acid gas from a Norblad generator was then passed through the liquid for about five hours. Quite a heavy white crystalline precipitate was obtained. This was thrown on to a porous plate and drained. It is very deliquescent. The concentrated solution of the crystals had a yellow colour. The mother-liquor changed colour from rose to orange while saturated with hydrochloric acid, due probably to the forcing back of the dissociation of the chloride by hydrochloric acid.

The mother-liquor was precipitated with ammonia and washed to remove chlorides, the precipitate dissolved in hydrochloric acid, concentrated, and again treated for two hours with hydrochloric acid gas in the freezing-mixture. Another heavy white crystalline precipitate was obtained, which gave a slightly yellow solution.

The spectra of the original solution and of saturated solutions of these two precipitates were compared by means of the small comparison spectroscope manufactured by Zeiss, of Jena, and described by Dr. Pulfrich in the *Zeitschrift für Instrumentenkunde*, October, 1900. No changes in the relative intensities of the various bands were visible.

Partial Decomposition of the Chromates.

Pattison and Clarke (CHEM. NEWS, xvi., 259) separated cerium from lanthanum and didymium by heating the normal chromates to 230° F. for some hours. The method when tried on this material gave no separation whatever. About 3 grms. of the oxides (R^{III} = 103·75) were dissolved in concentrated chromic acid, and the solution evaporated to dryness. The dry mass was heated for six hours at a temperature of about 200°, but decomposition merely on the surface was the only result, as could be told by the change in colour from the red of the chromate to the green of the chromic oxide. This same substance was then pulverised and heated for six hours to 250° with practically the same result. The substance was extracted with hot water, yielding a red solution and a greenish-black residue. The solution was evaporated to dryness, and was then heated to 300° for eight hours, but only very slight decomposition resulted. The insoluble greenish-black residue was again obtained from this portion. These two residues were treated with hot hydrochloric acid, and the green solution which was obtained was neutralised with ammonia, and precipitated with oxalic acid. Only a faint trace of the rare earth oxalates were thrown down.

Fractional Precipitation with Potassium Chromate.

The first to use this method in the separation of the rare earths was Gerhard Krüss (*Zeit. Anorg. Chem.*, iii., 92). This has since been modified by Moissan (*Comptes Rendus*, cxxii., 573), and in this form was employed by the authors on the material which gave R^{III} = 107·94. Krüss added a neutral solution of potassium chromate to a neutral nitrate solution of the earths, while Moissan used a sulphate solution of the earths, neutralising the liquid with ammonium hydroxide before each fractional precipitation with potassium chromate. The authors followed Moissan's directions carefully, but used a solution of the nitrates of the rare earths instead of the sulphates. This nitrate solution, amounting to 15 litres and containing about 140 grms. of the oxides, was first neutralised with ammonium hydroxide, and 1 litre of the solution of potassium chromate containing 97 grms. to the litre was added with continual stirring. The precipitation was carried on in tall glass cylinders of about 10 litres' capacity. The flocculent precipitate was allowed to settle, and the red supernatant liquid was removed by means of a syphon. The precipitate was washed with water until the wash-water gave no precipitate with ammonium hydroxide. The earths in the wash-water were recovered by precipitation with ammonium hydroxide, and this precipitate was dissolved in nitric acid to a neutral solution, and was added to the mother-liquor. This liquid was then again carefully neutralised with ammonium hydroxide, and was then treated with the same amount of potassium chromate solution as was used for the first fraction. This procedure was repeated four times, giving in all five fractions. The mother-liquor and wash-water from the last fraction were precipitated by ammonium hydroxide yielding fraction 6. With each succeeding precipitation the dilution of the solution was increased by about 1 litre.

Precipitate 1 was so small in amount as to render its separate treatment inadvisable; it was therefore united to precipitate 2, and the two were treated as one fraction. Each precipitate was dissolved in dilute hydrochloric acid, the chromic acid was reduced by adding alcohol and heating on the water-bath, and the resulting green solution was then nearly neutralised with ammonia, heated to boiling, and precipitated with a boiling solution of oxalic acid. In each case the precipitated oxalates were washed with a 1 per cent solution of hydrochloric acid, and were then dried and ignited. The resulting oxide was dissolved in hydrochloric acid. For the atomic weight determinations by the Gibbs method a portion of each of these solutions was precipitated with oxalic acid, and the oxalate was washed and dried.

Fraction 1 gave a pink oxalate, a deep orange oxide, and its saturated solution of a brownish-yellow colour showed

* *Journal of the American Chemical Society*, vol. xxiv., No. 5.

the same absorption bands as the original solution, but the bands were slightly stronger.

Fraction 2 was practically identical with fraction 1.

Fraction 3 gave a pink oxalate and an orange-yellow oxide somewhat lighter in tint than the oxide from fraction 2. Its saturated solution showed all of the absorption bands strongly.

Fraction 4 yielded an oxide somewhat lighter in colour than that from fraction 3, and its solution showed the bands plainly, but not as plainly as the preceding fractions.

Fraction 5 gave an oxalate of a pale pink colour and a very light yellow oxide. The saturated solution of its oxide was much paler in colour than the solutions of the preceding fractions, and while it showed all of the absorption bands they were much less distinct than in fraction 4.

Fraction 6 gave a perfectly white oxalate, and the oxide showed only the faintest tinge of buff colour, and its solution had a pale greenish-yellow tint. The absorption bands of this last solution were quite weak.

The atomic weights of the original material and the six fractions with fractions 1 and 2 united are as follows:—

Original, 107·94.

1 and 2.	3.	4.	5.	6.
133·54	126·13	114·04	97·83	94·01

Fractions 1, 2, 3, and 4 were then united, and were again fractionally precipitated by the chromate method. The atomic weights of the three fractions thus obtained were 145·34, 127·38, and 103·75. The middle fraction was then split up into two parts, one showing an atomic weight of 150·03, and the other 124·53.

Fractions 5 and 6 contain yttria with about 10 per cent of oxides having high atomic weights as terbia, erbia, and ytterbia. These two fractions comprise nearly half the material used in the fractionation. After 7 grms. of oxides with metal atomic weight of 107·07 had been extracted from them by this same process, the rest was set aside as yttria material. The two highest fractions R^{III} = 145·34 and 150·03 were also set aside for future use.

It is thus seen that fractionation with potassium chromate separates the earths of this group with considerable rapidity. It is especially serviceable as a means of obtaining yttria free from the other earths, this substance being separated in quite pure form at the end of a comparatively short series of fractions. The mixture of oxides in the first fractions is still a very complicated one, and further application of the chromate method does not appear to affect it appreciably.

Fractional Precipitation with Primary Potassium Oxalate.

The separation of the earths with this method proceeds but slowly, as is shown by the atomic weights given below. The method is a modification of the one used by Delafontaine for the separation of terbia from yttria and erbia. He added the solution of primary potassium oxalate to a solution of the rare earths until a point was reached at which another drop of the reagent caused a permanent precipitate. The liquid was then allowed to stand until the oxalates crystallised out. The solid substance was then removed, and crystallisation was repeated after the addition of more of the oxalate.

The authors used very dilute solutions in carrying on this method of fractionation, that of the primary potassium oxalate being about one-twelfth molecular, while the solution of the rare earths was diluted until the absorption bands were just visible with distinctness. The solution of primary potassium oxalate was made by dissolving in water molecular proportions of potassium oxalate and oxalic acid.

In order to be able to judge of the effect of fractional precipitation by this method upon the absorption bands of the solution, the precipitation was carried on before the spectroscope. The solution of the rare earths was transferred to a large crystallising dish about 20 c.m. in diameter, and this dish was placed directly before the slit of a Steinheil grating spectroscope. Light was furnished by a Linnemann zircon-disk lamp. The solution was kept in constant motion by means of a stirrer run by a Porter electric motor, and the solution of the primary potassium oxalate was added drop by drop from a burette. Before beginning the fractionation it is necessary to add a concentrated solution of the oxalate until a permanent precipitate just forms.

The first effect of the precipitation was to weaken the holmium bands, while all the bands decreased in intensity, the most persistent being the erbium band in the green. About 5 grms. of the oxides were obtained from fraction 1. Half of the mother-liquor from this fraction was then precipitated, and 6 grms. were obtained. This halving of the solution was made necessary because of the limited size of the dish, and concentration was undesirable because of the possible disturbing action of the more concentrated oxalate solution upon the earths. The mother-liquor from fraction 2 was again divided, and about 5 grms. of oxides were obtained in fraction 3. At the end of this third fraction all of the absorption bands had disappeared with the exception of the erbium band in the green, which was just visible. The mother-liquor from the last fraction amounting to one-fourth of the original solution was completely precipitated by the addition of the concentrated solution of primary potassium oxalate. Each of the precipitates was washed, dried, ignited, and dissolved in nitric acid. The oxalates of the first three fractions were pink, while that of the last one was white. The oxides of the first three fractions were orange-yellow in colour, while that of the last fraction had a pinkish tinge. All the solutions showed the rose-pink colour of erbium salts. In determining the atomic weights, a portion of each nitric acid solution was precipitated with ammonium hydroxide, and this precipitate was thoroughly washed with water to remove potassium salts. It was then dissolved in nitric acid to a barely acid solution, and precipitated with oxalic acid.

Original, 112·16.

1.	2.	3.	4.
118·80	116·71	111·08	102·53

The precipitates which are thrown down by these oxalate reagents are finely crystalline, and are consequently easily freed from the mother-liquor. Fractionation could probably be carried on with a fair degree of rapidity, and interesting results might be obtained. The separation was, however, too slow for the authors' purposes, and therefore no further trials have yet been made of it.

Fractional Decomposition of a Solution of the Mixed Nitrates by Means of an Electric Current.

About 120 c.c. of a solution of the nitrates (R^{III} = 107·94) were carefully neutralised with ammonium hydroxide. This solution was placed in a Classen platinum electrolysis dish, and was connected with the negative terminals of two cells of a storage battery. A platinum disk about 3 c.m. in diameter was used as the positive electrode. A low current of N. $D._{100}$ = 0·17 to 0·42 ampère at a potential difference between electrodes of 2·2 to 2·7 volts was passed into the solution for about twenty hours, the solution being kept neutral and at room temperature (about 21° C.). A white precipitate of the hydroxides was obtained at the cathode. A brownish deposit collected on the anode, but this was found to be lead dioxide due to a trace of lead present in the solution. The solution was poured off from the precipitate, the latter adhering to the dish sufficiently to make it possible to wash it without loss. The solution was evaporated to its original volume, and was again submitted to electrolysis, with a current of N. $D._{100}$ = 0·18 to 0·20 ampère at 1·8 to 1·9 volts for seven hours at 21° C. Again there was a small amount of precipitate formed. This being non-adherent, it was removed by filtration, and the wash-water and solution were again evaporated to the original volume and electrolysed. A current of N. $D._{100}$ = 0·17 ampère failed to produce any further precipitation after six hours, so the current was raised to N. $D._{100}$ = 0·33 to 0·42 ampère with a potential difference of 2·45 to 2·55 volts, and allowed to run for twenty-three hours. A further

white precipitate was obtained. These two precipitates were united for the second fraction; the atomic weight of the fraction was then determined, as was that of the first fraction and that of the residual solution. These three values compared with the original are as follows:—

Original, 107·94.

1.	2.	Residual solution.
120·22	116·61	106·20

If the decomposition voltages of the rare earths of this group were far enough apart to permit of a separation by means of electrolysis, the two fractions here should have atomic weights which differ more than do these. For if the earth or earth mixture which is thrown out of solution by a current of N. $D._{100} = 0.17$ ampère at a potential difference between electrodes of about 2·5 volts was different from that which is precipitated by a current of about twice the density, the difference should show itself in the atomic weights. It is evident therefore that, under the conditions here prevailing, the separation of the rare earths can only slowly be affected.

Fractional Precipitation with Magnesia Usta.

Muthmann and Rölig (*Ber. d. Chem. Ges.*, xxxi., 1719) separate cerium in the quadrivalent condition from lanthanum and didymium by precipitation with powdered zinc oxide, and then separate lanthanum from didymium by partially precipitating the hot concentrated solution with magnesia usta until the absorption bands of didymium have disappeared.

The effect of magnesium oxide as a precipitant was tried with a nearly saturated neutral solution of the chlorides of these yttrium earths ($R^{III} = 104·4$). Muthmann states that he stopped the precipitation when the didymium absorption bands had just disappeared. The authors found, however, that while a precipitate formed easily it would not settle sufficiently to enable the absorption bands to be observed.

The solution of the chlorides was heated to boiling, and while in vigorous ebullition powdered magnesia was added in small quantities, with constant stirring. The precipitate which soon separated was first washed by decantation, and was then washed upon a filter with hot water. The mother-liquor and the four wash-waters were evaporated nearly to saturation, and the precipitation was repeated. A second repetition of this procedure gave a third fraction, and the mother-liquor and wash-waters from this were then precipitated with oxalic acid for the fourth and final fraction.

Each of the precipitates was dissolved in hydrochloric acid, and the solution was concentrated. These four solutions showed practically no variations in absorption bands when examined with a direct vision spectroscope. The solution from the first precipitate was treated with ammonium chloride and ammonium oxalate as Muthmann recommends, and the resulting precipitate was washed by decantation with hot water containing 0·1 per cent hydrochloric acid, and was then washed with water alone. The precipitate was free from magnesia, but contained iron. The oxalates were at first non-crystalline, and settled very slowly, but the addition of the dilute acid caused them to separate in crystalline condition. It was found that, by adding oxalic acid to the neutral chloride solution of these precipitates, complete precipitation of the rare earths could be obtained, and this precipitate was free from magnesia, provided ammonium chloride was present. This method of purification was therefore used with the other fractions. The oxalates from all four of the fractions were ignited and dissolved in hydrochloric acid. Since the resulting solution contained traces of iron, portions of each fraction were freed from iron by re-precipitation with oxalic acid, and the atomic weight determinations were made with this purified material. The atomic weights and absorption spectra showed that very little change had been caused in the earth mixture by fractional precipitation with magnesium oxide.

(To be continued).

THE

RADIO-ACTIVITY OF THORIUM COMPOUNDS.

I. An Investigation of the Radio-active Emanation.

By E. RUTHERFORD, M.A., D.Sc.,
Macdonald Professor of Physics, McGill University, Montreal,
and
FREDERICK SODDY, B.A. (Oxon.),
Demonstrator in Chemistry, McGill University, Montreal

(Continued from p. 285).

Comparison of Intensity of Straight Line Radiation.

It was frequently of interest to obtain information about the intensity of the ordinary radiation correspondingly with measurement of emanating power. In order to do this rapidly and accurately, the following method was used. Fig. 2 shows the general arrangement. 0·1 grm. of the compound to be tested was reduced to fine powder and uniformly sifted over a platinum plate 36 sq. c.m. in area.

This plate was placed on a large metal plate connected to one pole of a battery of 300 volts, the other pole of which was earthed. An insulated parallel plate was placed about 6 c.m. above it, and the whole apparatus enclosed in a metal box connected to earth, to prevent electrostatic disturbance. The shaded portions in the figure represented insulators. A door was made in the apparatus so that the plate could be rapidly placed in position or removed. The current between the plates is observed in the usual way with the electrometer. The ratio of the currents for two substances is a comparative measure of their radio-activity. It is only possible to compare together with certainty substances of similar density and state of division,—a light, floury material will tend to give lower values than a dense powder.

If a substance gives off an emanation, the current between the plates increases with time. Under these conditions when the thoria is exposed in thin layers with a maximum of radiating surface, all but 1 or 2 per cent of the total effect is due to the straight line radiation; even when the effect due to the emanation has attained its maximum; this constitutes a very small percentage of the whole. This effect, however, may be to a large extent eliminated by taking the current between the electrodes immediately after the material is placed in the testing apparatus, or by passing a current of air between the electrodes to remove the emanation, and prevent it accumulating.

It is thus possible to compare intensity of radiations with an error not exceeding 1 or 2 per cent, and with great rapidity, and in these respects the electrical method is altogether superior to the photographic.

Comparison of Emanating Power.—The apparatus (Fig. 2) described for the comparison of radiations, can also be quite well employed for a comparison of emanating power. In this case, a thick layer of thoria (several grms.) is spread over the plate and covered with two thicknesses of ordinary paper. This has been found almost completely to stop the straight line radiation, whilst allowing the emanation to pass through unimpeded. The current is now measured when a steady state has been reached, due to the accumulation of the emanation. This takes some time, and draughts of air must be guarded against. For this reason, it is less convenient than the method first described, but the results obtained by the two methods are almost exactly the same. Thus a sample of "de-emanated" thoria which gave 12 per cent of the emanating power of the comparison sample by the first method gave 13 per cent by the second method, whilst a sample of oxide prepared from thorium oxalate gave 37 per cent and 39 per cent by the two methods respectively. The close agreement in the values by methods so completely different in character is a proof that the indications of the methods are worthy of a great degree of confidence.

The De-emanation of Thoria and the Regeneration of the Emanating Power.

The emanating power of thoria, as has been stated, is destroyed to a large extent by intense ignition. A closer study of this is the first step in the investigation of the phenomenon. Previous experiments had not succeeded in completely de-emanating thoria, although a reduction to about 15 per cent of its original value had been accomplished. A sample of this preparation which had been kept for two years had not altered from this value. An experiment was performed in which thoria was heated to the highest temperature which could be safely employed with platinum vessels: (1) in a thin layer in a large platinum dish, and (2) in bulk in a small platinum crucible placed inside the dish. The two were heated together by means of a powerful gasoline furnace for one hour. The temperature was such that the fireclay walls fused, and the pipeclay of a triangle showed signs of having been softened. It was found that the sample that had been heated in a thin layer in the dish retained about 16 per cent of its original emanating power, whilst the other sample retained about 8 per cent. There is thus no advantage in heating in thin layers, in fact, rather the reverse, for the sample showing 16 per cent again heated to a slightly lower temperature for half-an-hour in a small crucible was reduced to 12 per cent.

In another experiment, a small platinum crucible filled with thoria was heated for half-an-hour in a small furnace by a large blowpipe and powerful pair of bellows. Some asbestos-wool had completely fused on the outside of the crucible, and the temperature was probably but little lower than in the previous experiment. This sample also retained about 8 per cent of its emanating power. No further attempt has yet been made to completely destroy the emanating power.

A small quantity of thoria heated in a platinum crucible in the open over an ordinary small sized blowpipe and bellows for five minutes retained about 45 per cent of its emanating power. The effect of time as well as of temperature was studied by heating about equal quantities in a platinum crucible over an ordinary Bunsen burner for different periods.

Heated 10 minutes	Emanating power	= 61 per cent
„ 1 hour	„	= 59 „
„ 24 hours	„	= 42 „

It thus appears that there is a large and practically sudden decrease of emanating power for each temperature above a red heat, followed by a very gradual decrease with time when the temperature is maintained; thus five minutes on the blowpipe, whilst much more effective than the same time at the temperature of the Bunsen burner, produced rather less effect than twenty-four hours at the latter temperature.

Effect of Moisture.—The next point to be examined was whether the loss of emanating power could be attributed to a loss of water and desiccation of the thoria by ignition. A sample of de-emanated thoria (retaining about 14 per cent) was placed in the middle of a Jena glass tube, one end of which was closed and contained water, the other end being drawn out to a jet. This was supported in a powerful tube furnace in a sloping position, and the part containing the thoria heated to the highest possible temperature, while a slow current of steam from the water at the end was passed over it, escaping by the jet. When all the water had evaporated, the jet was drawn off and the tube allowed to cool in an atmosphere of steam free from air. The thoria, on testing, was found to have been lowered in emanating power to about 7 per cent. The further heating had thus reduced the emanating power without the steam having at all regenerated it.

In the next experiment, the reverse process was tried. Two exactly parallel processes were carried out for ordinary thoria possessing the normal amount of emanating power. In the first, it was heated in a porcelain tube in the tube furnace for three hours, while about 500 c.c of water were distilled over it from a retort. In the second, another quantity of thoria was heated in exactly the same way for the same time, only a current of well dried air was substituted for the steam. The result was conclusive: each sample had had its emanating power reduced to exactly the same amount; that is, about 50 per cent of the original.

These experiments prove that water vapour exerts no influence either in de-emanating thoria or in effecting a recovery of its lost emanating power.

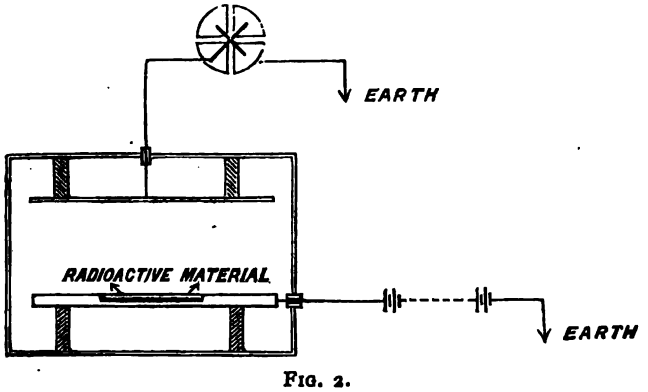


FIG. 2.

The Re-generation of the Emanating Power by Chemical Processes.—The task of subjecting de-emanated thoria to a series of chemical changes to see if it would recover its lost emanating power was then undertaken.

It may first be mentioned that thoria which has been subjected to ignition has changed very materially in chemical and physical properties. The pure white colour changes at temperatures corresponding to the first stages of de-emanation to a light brown, and after subjection to the very highest temperatures to a pure pink. At the same time, as has been observed before, the solubility of the substance in sulphuric acid is greatly diminished. A part always obstinately refuses to dissolve, even after long and repeated boiling with the concentrated acid, although this part is diminished by each successive treatment and appears to be in no way different from the rest of the substance. No difference, however, occurs in the readiness with which chlorine attacks it when intimately mixed with carbon. The formation of the chloride by this method is the easiest way of dissolving ignited thoria.

Two quantities of the same de-emanated thoria were converted, the one into chloride and the other into sulphate, by the usual methods, and from each of these the oxalate was formed by precipitation of the acid solution with oxalic acid. Parts of the oxalates were then converted into oxides by heating over the Bunsen burner. In both cases there was a very marked recovery in emanating

power; the oxide obtained from the sulphate had about 40 per cent, that from the chloride about 55 per cent, whereas the de-emanated thoria from which they were both produced had about 13 to 14 per cent of the emanating power of thoria. The oxalates from which the oxides were formed each had about 11 per cent of the power, and in converting them into oxides it was ascertained by a direct trial that too high a temperature had been employed and the thorium oxide had suffered partial de-emanation. At this time also, it was beginning to be realised that the emanating power was a quantity which varied, not only with the nature of the chemical compound, but also for the same compound very materially with its previous history. Thus the oxide from the oxalate does not possess as a rule so great an emanating power as that used for comparison, which would account for the above result. The following two exactly parallel experiments were therefore made—the one with ordinary, and the other with de-emanated thoria possessing 9 to 10 per cent of the emanating power of the first. Each was converted to chloride in the ordinary way, by mixing with sugar solution, carbonising, and igniting the mixture of oxide and carbon so obtained in a current of dry chlorine. Each sample was then treated with water, the thorium precipitated as hydroxide with ammonia, and the hydroxides washed and dried at 110°. The hydroxide prepared from the de-emanated thoria possessed 128 per cent, that from the ordinary thoria 108 per cent of the emanating power of ordinary thoria, when tested immediately after drying. Now a sample of hydroxide previously obtained had shown no less than three times the emanating power of ordinary thoria. The specimens were therefore tested again after having been kept for four days in loosely corked tubes. They now showed 157 per cent and 139 per cent respectively. The emanating power was thus increasing, so both specimens were exposed side by side in open watch-glasses under a sheet of glass to keep off the dust. The result is again conclusive:—

	From de-emanated ThO_2.	From ordinary ThO_2.
	Per cent.	Per cent.
After nine days	253	253
After three more days ..	259	259

Thus the process of de-emanating thoria by ignition does not irretrievably destroy the emanating power, for after solution and re-precipitation no difference whatever exists in the emanating power between ordinary and de-emanated thoria.

The results also bring out another point,—the marked effect of time and exposure to air in increasing the emanating power of thorium hydroxide. This will be examined more fully later.

A fair conclusion from these experiments is that the cause of the emanating power is not removed by ignition, but only rendered for the time being inoperative.

Radio-activity of De-emanated Thoria.—The "straight line" radiation of thoria, de-emanated as completely as possible by ignition, was compared with that of ordinary thorium oxide by the method described. It was found that within the limits of error no difference whatever could be detected between them. This result serves to bring out the fact that the power of thoria to give an emanation is independent of its power to give a direct radiation.

Is the Emanating Power a Specific Property of Thorium?

Having shown that the de-emanation of thoria by the processes described consists rather in a temporary obliteration of the effect than in a removal of the cause producing it, the next question to be considered is whether it is possible to remove from thorium compounds by chemical methods any constituent to which the property of emanating power can be traced.

The thoria used in the investigation is that supplied by Messrs. Eimer and Amend, of New York, and is obtained from monazite sand by a secret process. It, of course, does not consist of pure thoria, although from superficial investigation it appears to be of excellent quality. There is a small quantity of a substance present which can be precipitated by sodium phosphate after removal of the thorium as hydroxide by ammonia, the nature of which is at present under investigation. The most noticeable impurity is about 1 per cent of thorium sulphate. Careful washing completely removed this impurity, and the emanating power of the washed sample was identical with the ordinary. The impurity may therefore be disregarded for present purposes.

Emanating power is not confined to thorium from any particular source. Orangeite and thorite from Norway both possess it, as well as monazite sand from Brazil. A specimen of thoria prepared from orangeite by the ordinary processes possessed about the same emanating power as that obtained from monazite sand by the secret process.

A quantity of thorium oxide was converted into the anhydrous sulphate and dissolved in ice water. The temperature was allowed to rise, and the hydrated sulphate precipitated in four fractions, a fifth being obtained by evaporation of the mother-liquor to dryness. These showed no marked difference in emanating power among themselves. The first fraction was dehydrated and again submitted to fractional precipitation as hydrated sulphate. The first fraction of the new series—designated fraction AA—was then compared in the following manner with the mother-liquor fraction of the first series—designated as fraction E. Both were dehydrated, dissolved in water, and precipitated as hydroxide by ammonia, washed and dried under the same conditions, and compared together at regular intervals with a comparison sample of ordinary thoria.

	Fraction AA.	Fraction E.
	Per cent.	Per cent.
At first	203	200
After one day	240	249
After thirteen days	316	321
After forty-three days	352	372

The differences are too small to afford any indication of separation of the emanating material.

The straight line radiations of the two fractions tested in the apparatus (Fig. 2) also proved to be identical.

It was obviously useless trying any further fractionations by this method. Since there was no appreciable difference in either property in the fractions tried, there was nothing to be gained in a further repetition. These results completely accord with those of Sir William Crookes (*loc. cit.*), with which, however, we were not acquainted until after our own experiments had been performed.

Another method for the purification of thoria, employed by Dennis (*Journ. Am. Chem. Soc.*, 1896, xviii., 947), the precipitation of the hydroxide by potassium azoimide, was next tried. The latter reagent was prepared by Thiele's method (*Annalen*, 1892, cclxx., 1) from diazoguanidine nitrate. Hydrazoic acid partially neutralised with potash precipitates thorium hydroxide from the boiling solution of a thorium salt. This hydroxide, compared with a sample which had been precipitated with ammonia in the ordinary way, showed similar emanating power.

These results, which fail to give any indication of a separation of the emanating material by chemical means, taken in conjunction with those already described in the preceding section on the regeneration of the emanating power in de-emanated thoria, certainly seemed to point to the conclusion that the power of giving an emanation is really a specific property of thorium. Recent results, which will be given in the last section, put the question in a fresh light.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, Wednesday, May 28th, 1902.

Prof. Meldola, F.R.S., Vice-President, in the Chair.

Messrs. J. W. Peck, J. C. Crocker, and J. Kewley were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Edwin T. H. Bucknell, Ebley House, Parsonage Street, Dursley, Glous.; Francis E. Francis, 8, Manilla Road, Clifton, Bristol; George Paton Pollitt, Ph.D., St. Silas Road, Blackburn, Lancs.; Harold James Roast, 3, Manor Park Road, Harlesden, N.W.; Edward J. Wheeler, Albany, State of New York, U.S.A.

Of the following papers, those marked * were read:—

*81. "*Taxine, the Alkaloid of Yew.*" By T. E. Thorpe, C.B., F.R.S., and G. Stubbs.

The investigation has been carried out on autumn-gathered leaves of male and female trees of the species *Taxus Baccata*. The alkaloid was extracted by digesting the powdered, air-dried leaves with 1 per cent sulphuric acid for five or six days. The acid liquid was strained and pressed from the leaves, and at once, without concentration, rendered alkaline, and extracted with ether. Taxine was obtained in the form of very fine, glistening particles, by crushing down the residue from the ether extract. It gives precipitates with most of the alkaloidal reagents, and colour reactions with strong sulphuric acid alone, and when this reagent is mixed with nitric acid, molybdic acid, or chromic acid.

Taxine is very susceptible to change. At least two substances result from the action of dilute acids upon it, but owing to lack of material the authors have not yet completed their investigation of these products.

Several salts have been prepared and analysed. Two compounds with gold chloride have been obtained which have the formulæ $C_{37}H_{52}NO_{10},HAuCl_4$ (m. p. 72·5°) and $C_{37}H_{52}NO_{10},AuCl_3$ (m. p. 132—134°) respectively. The methyl iodide compound, $C_{37}H_{52}NO_{10},CH_3I$ (m. p. 121°), has been prepared as a white, amorphous powder by mixing benzene solutions of the alkaloid and methyl iodide.

Although the analytical figures deduced, both for taxine and its salts, are in substantial agreement with the formula, $C_{37}H_{52}NO_{10}$, given by Hilger and Brande (*Ber.*, 1890, xxiii., 464), the authors are not of opinion that this formula is definitely established.

The alcoholic extract of yew, amounting to about 26 per cent of the dry leaves, is at present under examination.

Discussion.

Prof. Dunstan remarked that the authors had confirmed the accuracy of previous work on the properties of taxine, and he hoped that they would now be able to go further and throw some light on its chemical structure. It had not been yet fully proved that yew-poisoning was due to the presence of taxine, and indeed even the exact conditions under which yew becomes poisonous did not appear to have been determined with certainty.

Mr. Golding said that in the year 1893 he had assisted Mr. Dymond in an investigation of the alkaloid of the yew. They found that drying the leaves at 100° destroyed the alkaloid.

The extraction of the alkaloid was made from bruised, green, freshly-gathered leaves by Marmé's process, which was repeated several times to purify the alkaloid. Further purification was effected by adding light petroleum to the ether solution, the fractions thus obtained giving the characteristic reactions of taxine.

When dry hydrogen chloride was passed into a solution of the alkaloid in anhydrous ether, and a crystalline precipitate was formed, which soon changed to a dark brown paste, phenomena also observed by Hilger and Brande.

Cases were on record of animals eating yew with impunity, and a large number of the cases which had proved fatal were due to the eating of *withered* branches.

Dr. Voelcker pointed out that the uncertainty with regard to poisoning by yew extended to the kind of animal eating it as well as the condition of the leaves. A good deal also depended on the conditions under which it was eaten; whether, for example, much grass or other food was eaten along with it. He thought that it was very desirable that the other substances which the authors had obtained should also be studied, but regarded it as essential that the yew examined should be freshly gathered, and not kept for some considerable time as in the case of that employed by the authors.

Mr. Stubbs, in reply, said that no work had been done to ascertain if the alkaloid contained methyl groups. He did not think that the substance had been produced by the action of the acid used for extraction upon a glucoside. The material employed had been dried in air, at a temperature not exceeding 60°. They had obtained the same amounts of alkaloid from the leaves both when freshly gathered and after they had been dried.

*82. "*The Sampling of Soils.*" By J. W. Leather.

Experiments were made in India to determine the accuracy of the auger method of sampling soils, and the agreement between the samples was tested by determinations of the total nitrogen, the available phosphoric acid, and the available potash. The results showed that in most cases the agreement was very good between samples, but that in some cases the divergence exceeded 1 in 20, an amount which the author considered too great.

The amounts of phosphoric acid in some of the Rothamsted wheat soils, as found by Dyer, were considered with a view to determine the accuracy of the method of sampling which was there employed. The author considered that such a comparison indicated the Rothamsted method to have yielded as good results as the auger method. Both, however, fall short of what is expected in taking samples of commercial products, and it was urged that the newer methods of soil investigation demand more accurate methods of sampling.

Discussion.

Dr. Dyer said that it seemed obvious that a sample drawn from many parts of the field was more likely to be accurately representative than a sample drawn from one or few places. Nevertheless, it seemed to him that Dr. Leather's analytical results, obtained on samples drawn from different parts of the same field, instead of indicating differences in the samples, appeared really to indicate that both the sampling and the analytical work were excellently performed. Some of the analytical figures regarded by Dr. Leather as discrepant, he (the speaker) should himself regard as being in very good agreement. As to the results of his determinations of phosphoric acid in the Rothamsted wheat soils, which Dr. Leather had quoted, it must be remembered that, whilst the samples analysed represented only the first nine inches of soil, the phosphoric acid removed in the crops might in the case of wheat, on many plots, be drawn to a considerable extent from the subsoil; and that the influences of vegetation might considerably affect the distribution of soil constituents in the various layers of the soil and subsoil. The "original" phosphoric acid of Broadbalk field, as calculated back from the present contents, and known additions and removals, showed, on the whole, as Dr. Leather had pointed out, a fair uniformity, though there were some discrepancies. It was, however, quite probable that the percentage of phosphoric acid in the surface soil was originally by no means uniform on the different plots. Certain of the plots had long behaved somewhat anomalously, indicating some inherent physical or chemical differences in the soil or subsoil.

Dr. Voelcker was inclined to agree with Dr. Dyer, and to regard the results which Dr. Leather had obtained as being very satisfactory, and as agreeing well with one another. It would hardly be a matter for surprise to those

who had followed the work at Rothamsted, and who knew the infinite pains which the late Sir Henry Gilbert always took to ensure accuracy in every detail, to learn that Dr. Leather's estimate of gains and losses showed that there could not be much amiss with the Rothamsted method of soil-sampling. Still, it was obvious that the more places in which borings of a soil were taken for sampling purposes the more representative would that sample be.

There was considerable difference between soil samples taken as at Rothamsted (where at least three holes were selected on each plot), and the block of soil ordinarily sent by a farmer when he wished his soil analysed. In the latter case, no doubt, the method Dr. Leather suggested would give a much more representative sample.

The difficulty was in the case of soils of stony character, or where lumps of chalk occurred. The auger might very readily, by pushing these stones or lumps aside, or by including them in the boring, ultimately cause a considerable difference in the analysis.

Mr. Hall said that for some years he had been using an auger for taking samples in the Soil Survey of Kent and Surrey they were carrying out. They used a cylindrical auger one foot long, two inches in diameter, with a slot one inch in breadth, the sides of the slot and the bottom of the cylinder being each sharpened to a cutting edge. The worm auger was only used for exploring the subsoil to some depth. The auger was undoubtedly most useful for securing a fair sample, especially in field work, where one did not want to be burdened with a great weight of soil; but there were many soils on which it could not be used, they were either too hard, too stony, or too loose to remain in the tool. The steel box employed at Rothamsted could be made to give the most accurate results, but it was not wise to lay down any hard and fast rule as to which was the best tool. The analyst must examine the soil, and then use his judgment.

As to Dr. Leather's figures, not only did they support his opinion that fair samples could be drawn with the auger, but they showed that he must have been working with an extremely uniform soil, probably of alluvial origin. In England samples taken from adjoining fields on the same formation would vary between far wider limits than those shown by Dr. Leather's figures.

Dr. Leather, in reply, said that so far as stones are concerned, the determination of their amount would naturally demand that comparatively large portions of soil should be taken, but this consideration would not apply to the examination of the fine earth. Of the Indian soils examined, that at Cawnpore is alluvial, but the geological history of the Poona land is uncertain.

*83. "*Some Excessively Saline Indian Well-waters.*" By J. W. Leather.

The composition of some well-waters from the Muttra District, United Provinces, India, was given in detail. These waters contain from 200 to 2000 parts of salt per 100,000 of water. About one-half of the saline matter consists of sodium chloride; the amount of carbonate is about 20 to 30 parts; the nitrate varies from nothing to 250 parts; very large amounts of sulphate were present in some of the samples. In three cases, there was an excess of lime over that required to combine with the nitric, carbonic, and sulphuric acids, thus proving the presence of calcium chloride.

Some of these waters are used for irrigating crops. The soil of the district is open, the drainage conditions are good, and no salts accumulate in the surface soil. These waters provide an indication therefore of the maximum strength of solutions in which plants can grow, a point which is of importance in connection with the alkali lands of America and the Usar lands of India. It is found practically that, of these well-waters, those may be employed for irrigating crops which do not contain much more than about 500 parts of salts per 100,000, or 0·5 per cent.

The amount of salts in Usar or alkali land is usually stated in parts in 100 of soil, and it is found in practice that soils containing much more than 0·1 per cent are infertile. Immediately after irrigation, such a soil would contain a solution of about 0·33 of salts per 100 of water, but after the lapse of a few days, the proportion of water decreases to 10 or 15 per cent, and in this state the above mentioned solution would become concentrated to about 1·0 or 0·66 per cent respectively. These figures correspond practically with what is known about the well-waters.

Discussion.

Mr. J. Spiller said that in the year 1865 he had occasion to examine some remarkable products obtained from a highly saline spring which discharged itself into Lake Lohhar, India. The water left on evaporation 8·25 per cent of solid residue, which consisted roughly of two parts of common salt to one of sodium carbonate. A local attempt at a separation of these resulted in a purified product, which had the following composition:—

Sodium carbonate	75·0
„ chloride	0·6
Calcium carbonate	1·9
Sand and ferric oxide	2·7
Water, nearly	20·0

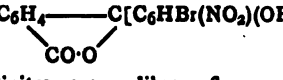
There was no sulphate, bromide, borate, or nitrate.

*84. "*Nitrobromo-derivatives of Fluorescein.*" By J. T. Hewitt and A. W. G. Woodforde.

The non-fluorescence of alkaline solutions of nitro-derivatives of fluorescein was explained by the assumption that the alkaline salts of orthonitrophenols have the metal attached to the nitroxyl and not to the hydroxyl group (Hewitt and Perkins, *Trans.*, 1900, lxxvii., 1324). With regard to the position of the nitro-groups in dinitrofluorescein, it was assumed that these occupied positions 2 and 7. (The numbering of the fluoran ring corresponds to Richter's numbering in the xanthene nucleus).

Additional support was lent to this view by the isolation from the product of alkaline fusion of a small amount of material having a melting-point of 114°. Since 4-nitroresorcin melts at 115°, the substances were supposed to be identical, and hence positions 2 and 7 were assigned to the nitro-groups in the fluoran nucleus. It is now shown, however, that this view must be discarded; by adopting suitable precautions, 2-nitroresorcin may be isolated in considerable quantity, as had been previously demonstrated by Matras; the authors' thanks are due to M. Reverdin, of Geneva, for calling their attention to this work.

Dinitrofluorescein is thus 4 : 5-dinitrofluorescein; acted on by bromine, 4 : 5-dinitro-2 : 7-dibromofluorescein results. This substance closely resembles the original dinitrofluorescein; it dissolves in alkalis with a brown colour, but on warming a blue solution is obtained from which acids precipitate the hydrate—



4 : 5-Dinitro-2 : 7-dibromofluorescein has been further characterised by its *diacetyl* derivative, $C_{20}H_6Br_2N_2O_9(C_2H_3O)_2$, m. p. 276°; *dibenzoyl* derivative $C_{20}H_6Br_2N_2O_9(C_7H_5O)_2$, m. p. 315°; *sodium* salt, $C_{20}H_6Br_2N_2O_9Na_2.2H_2O$.

Dibromofluorescein, $C_{20}H_{10}Br_2O_5$, obtained by Bayer by the limited action of bromine on fluorescein, is evidently also a 4 : 5-substitution derivative, since nitric acid produces from it a dinitro-derivative quite distinct from the 4 : 5-dinitro-2 : 7-dibromo-compound. The *benzoyl* derivative, $C_{20}H_8Br_2O_5(C_7H_5O)_2$, melts at 240—244°. 2 : 7-*Dinitro*-4 : 5-*dibromofluorescein* may be obtained either by the action of nitric acid upon 2 : 7-dibromofluorescein or of bromine upon 2 : 4 : 5 : 7-tetranitrofluorescein. This substance dissolves in alkalis with a magnificent purple colour not altered by boiling. Nitro-groups only render the pyrone ring unstable if in the ortho-position relatively to its oxygen atom; this is also shown by the action of ammonia, which reacts with 4 : 5-dinitro-2 : 7-dibromofluorescein, apparently in a similar manner to that ob-

served by Reverdin in the case of dinitrofluorescein, whilst with 2:7-dinitro-4:5-dibromofluorescein it merely produces an ammonium salt.

The *diacetyl* derivative, m. p. 215°; *dibenzoyl* derivative, m. p. 301°; and *sodium* salt of 2:7-dinitro-4:5-dibromofluorescein have been prepared.

(To be continued).

THE ROYAL SOCIETY.

Conversazione at Burlington House.

A very well attended and successful Conversazione, to which ladies were also invited, was held at the Society's Rooms, Burlington House, on Wednesday evening last, June 18th. The Fellows and guests were received by Sir William and Lady Huggins, and many distinguished visitors were present.

Among the interesting and varied exhibits we may mention an Apparatus for Liquefying Hydrogen, exhibited by Dr. Morris Travers. Hydrogen, when compressed at the ordinary temperature and allowed to expand, becomes warmer, while air under the same conditions becomes colder; at temperatures below −80° C., hydrogen becomes an imperfect gas, in the same sense as air, and undergoes cooling on free expansion (Joule-Kelvin effect). The gas, under a pressure of 120−150 atmospheres, passes through coils in the interior of the apparatus; these coils are cooled in solid carbonic acid and alcohol (−78·5° C.), in liquid air (−185° C.), and in liquid air boiling under reduced pressure (−200° C.). It then enters a regenerator coil, and expanding at a valve at the bottom is partially liquefied. The liquid collects in a vacuum-vessel at the bottom of the apparatus; the unliquefied gas passes upwards through the regenerator coil, cooling the gas it contains, and returns to the compressor.

Prof. W. Ramsay showed an attempt to reproduce an Aurora Borealis. The spectrum of the aurora borealis has been shown to contain lines due to the presence of krypton; the great majority of the lines, if not all, are coincident with those of the krypton spark spectrum. An electrode-less discharge in air gives a spectrum in which the leading green line of krypton, 5570·5, is distinctly visible at low pressures. This discharge can be deflected by a magnet, sending out streamers in the lines of magnetic force. The main phenomena of the aurora are thus reproduced.

Prof. H. L. Callendar showed an Apparatus for Determining the Mechanical Equivalent of Heat, by which measurements of one part in a thousand can be obtained; he also showed some vacuum-jacket calorimeters embodying the most recent improvements.

The West Indian Volcanoes Committee showed some specimens and photographs illustrating the fall of volcanic dust at Barbados on May 7th and 8th, 1902; while Mr. Henry Crookes also showed specimens and photographs of a similar nature, viz.:—

(1). Volcanic Dust from La Soufrière, St. Vincent. This dust erupted from La Soufrière, and fell upon Barbados, May 8th, 1902. It is estimated that from 2,000,000 to 3,000,000 tons fell in seven or eight hours.

(2). Volcanic Dust collected from the steps at Roxborough, Grenada, about 90 miles from La Soufrière, May 8th, 1902.

(3). Volcanic Dust from the explosion of Mont Pelée, Martinique. This specimen is of special interest, as it was collected on the deck of the S.S. *Roddam*, Captain Freeman, on her arrival at St. Lucia. The *Roddam* was the only ship that escaped from St. Pierre, and many of her crew were burned to death.

(4). Micro-photographs of the above specimens of volcanic dust.

(5). Microscopic Slides of the same viewed with ordinary, and with polarised light.

(6). Scoria, from the eruption at Krakatoa in 1883, taken from the sea about 400 miles from the volcano.

The microscopic examination of the dust from the deck of the *Roddam* shows that it consists of nearly equal amounts of mineral and rock fragments, from about one five-thousandth to one-hundredth of an inch in diameter, but commonly seven- or eight-thousandths of an inch. The minerals are felspar (generally labradorite), augite, and hypersthene; the rock fragments, a pale brownish-grey scoria, in one or two cases red, generally opaque, and with traces of a brown glass. The dust from La Soufrière from Barbados is of a very similar character, each representing a rock of the same species, viz., that called "Hypersthene-andesite."

Dr. G. Johnstone Stoney showed some Experiments Exhibiting Interference between Portions of Light from Independent Sources. In one experiment a spectrometer is employed, in the other a microscope. In both, spectra will be seen. These are produced because interference directs the light with different intensities towards the various directions; and in the present experiments, essential interfering portions of the light are taken from widely separated parts of a flame, and therefore come from independent sources.

During the evening demonstrations by means of the electric lantern were given in the Meeting Room by Prof. W. M. Flinders Petrie on Early Civilisation in Egypt; by Mr. J. Y. Buchanan, who showed a series of Lantern Slides, illustrating the performance of M. Santos Dumont's dirigeable balloon and the accident to it on February 14th, 1902; and by Prof. E. B. Poulton, on Recent work upon Protective Resemblance and Mimicry in Insects, illustrated by three-colour slides.

PHYSICAL SOCIETY.

Ordinary Meeting, June 13th, 1902.

Prof. S. P. Thompson, President, in the Chair.

The meeting was held in the National Physical Laboratory by the kind invitation of the Director.

Dr. Glazebrook made a brief statement, and said that the task of fitting up an ordinary house as a physical laboratory had been a difficult one, much more difficult than that of designing a new building. The general scheme of the laboratory is such that the main departments, including those of electricity, thermometry, and chemistry, occupy the principal rooms in four of the wings, together with the portions of the basement most nearly adjoining, while the first floor and a number of small rooms are fitted up as general laboratories. An additional building has been constructed for carrying on the work of the engineering department. Electric current is supplied by fifty storage-cells and distributed by bare wires carried by porcelain insulators. From the main switchboard and four subsidiary switchboards, circuits are led to almost every room in the building, and to these any voltage from 2 to 110 can be applied by making a suitable connection.

The meeting then adjourned, and the Fellows were allowed to inspect the laboratory.

Thermometry.—Dr. Harker showed apparatus for testing thermometers at high and low temperatures, including:—

(i.). An oil-bath. Temperatures 100° C. to 240° C.

(ii.). A bath of fused nitrates of sodium and potassium. Temperatures 240° C. to 550° C.

(iii.). Electric ovens. Temperatures up to 1250° C.

(iv.). A vacuum vessel with suitable cooling agents.

High-temperature mercury thermometers, platinum thermometers, and thermopiles were shown in operation, and a demonstration was given with the Hampson apparatus for the liquefaction of air.

Electricity.—Mr. Campbell and Mr. Melsome showed:

(i.). A Kelvin electrostatic voltmeter with a long scale and arrangements for calibrating against a Clarke's cell.

(ii.). A Kelvin current-balance,

(iii.). Apparatus for determining the capacity of the standard air-condensers of the British Association.
(iv.). Apparatus for tests on iron by the ballistic method.
(v.). Ewing's permeameter.
(vi.). Bridge for resistance measurements.
(vii.). Methods for measuring a small resistance:—
(*a*) Thomson's double bridge.
(*b*) Potentiometer method.
(*c*) Differential galvanometer method as described by Crawley.

Electrical Standards.—Mr. Smith showed the British Association standards of resistance, and the standard cells in a room maintained at a constant temperature by a gas-fire and a regulator.

Terrestrial Magnetism.—The latest form of unifilar magnetometer, designed for the Indian Survey, together with some recent magnetic and seismographical charts were shown by Dr. Chree. Mr. Watson's magnetographs, arranged for the special magnetic observations in connection with the Antarctic Expedition, were exhibited.

Microphotography.—Dr. Carpenter exhibited the Zeiss microphotographic outfit, and showed some microphotographs of steel.

Metrology.—The following apparatus was shown by Mr. Keeling:—
(i.). A Whitworth measuring machine.
(ii.). A Pratt and Whitney measuring machine.
(iii.). A dividing engine by the Société Genevoise.
(iv.). A simple comparator.
(v.). A comparator by the Société Genevoise.

Photographic Lens-testing.—Mr. Hugo exhibited the Kew apparatus for testing photographic lenses.

The chemical laboratory, the metallurgical laboratory, and the main switchboard room were also open for inspection.

In the *Engineering Laboratory* Dr. Stanton and Mr. Jakemann showed the steam turbine and dynamos, the gauge-testing apparatus, the experimental testing machine, and the other machines which have been installed.

NOTICES OF BOOKS.

Grundriss der Qualitativen Analyse. By Dr. Wilh. Böttger. Leipzig: Wilhelm Engelmann. 1902.

The rapid spread of the ionic theory, in spite of many opponents, creates a demand for text-books written in accordance with new ideas, and adapted for students' use, and there seems to be no fear but that this want will be amply supplied. This book gives a thoroughly satisfactory introduction to the study of qualitative analysis from the ionic point of view, and will prove specially useful to those who are using Ostwald's "Grundlinien der Anorganischen Chemie," to which references are constantly made. Innovations in matters of detail are not wanting, but will in nearly all cases justify themselves; such theoretical discussions as are introduced are carefully worded, though perhaps occasionally the student may find them a little above him. The reactions of kations occupy the first part of the book; here the ordinary order of procedure is reversed, and the student studies first the separation of the metals of a group, and then passes on to the examination of the characteristic reactions of each metal. It is doubtful whether this plan is an improvement on the more usual one of deducing as far as possible the method of separation from the previously observed reactions of the different individual members of a group; however, it would be easy for the teacher to modify this arrangement at his discretion. The anions are grouped more systematically than is usually the case, and if Dr. Böttger's method is strictly observed no difficulty should be found in the detection of acid radicles. The book concludes with directions for the detection of the rarer elements.

Die Elektrolyse des Wassers. By Viktor Engelhardt. Halle-a-S.: Wilhelm Knapp. 1902.

This volume is the first of a series of monographs on applied electrochemistry, to be issued quarterly. The aim of the authors of these monographs is to be the production of complete and trustworthy accounts of special branches of the rapidly developing science of electrochemistry; each volume will treat of the historical development of its special subject, and will endeavour to give a good insight into the most recent advances relating to it, and the hope is expressed that both the practical man and the student may derive instruction from them. This first volume deals with the electrolysis of water. Distinction is made between those methods and apparatus suitable for laboratory work and the lecture table, and those better adapted for use technically on the large scale. Patent specifications have been carefully searched, and are freely quoted and summarised, and useful commercial data, such as the cost of building the plant and carrying on the processes, are given in considerable detail. The issue of future volumes on such subjects as the electrometallurgy of iron, the preparation of ozone, and the electrolysis of organic compounds will be awaited with interest.

Catalogue of the Educational Collection of Minerals Belonging to the West Ham Municipal Technical Institute. Compiled by H. A. Auden, D.Sc., M.Sc., Head of the Chemical Department.

This catalogue has been compiled with a view to illustrate the systematic grouping of mineral specimens.

Each opening of the catalogue is divided into ten columns, giving the specimen number, name and composition, system, synonyms and varieties, isomorphic grouping, &c., colour, similar minerals, associated minerals, hardness, and specific gravity.

The classification adopted is based on that of Klockmann, and is clear, comprehensive, and systematic.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxiv., No. 21, May 26, 1902.

Synthesis of certain Petrols. Theory of Formation of Natural Petrols.—Paul Sabatier and J. B. Senderens.—The author succeeds in synthesising several petrols, and deduces from this a very simple explanation of the formation of natural petrols. In the depths of the earth the alkaline and alkaline earth metals in the free state are widely distributed besides their carbides. Water on coming in contact with these metals causes the evolution of hydrogen, whilst, in contact with the carbides, acetylene is evolved. These two gases in variable proportions come in contact with the various metals found in nature (nickel, cobalt, iron, &c.) in a finely divided state, and certain reactions occur by which the various petrols are furnished.

Certain Physical Properties of Hydrogen Telluride.—MM. de Forcrand and Fonzes-Diacon.—The authors prepare pure hydrogen telluride and determine the following constants:—

T (temperature of ebullition), 0° C. or 273° abs.
T' (temperature of fusion), −48° C. or 225° abs.
D (liquid density), 2·57.
PM/D (molecular volume), 49·75.

In the case of H_2S and H_2Se the constants are as follows:—

	H_2S.	H_2Se.
T	211·4°	231°
T'	187°	209°
D	0·86	2·12
PM/D	39·53	38·20

The molecular volume differs considerably from the almost identical values found for the other two gases, and still more from that of water (18·82).

Preparation and Properties of Chloro-, Bromo-, and Iodo-sulphobismuthites of Copper.—Fernand Ducatte.—The presence of a halogen element in definite proportion which the author has found in the products resulting from the action of lead chloride, bromide, or iodide on bismuth sulphide, Bi_2S_3, is not an isolated fact, but products are obtained analogous with cuprous chloride, bromide, and iodide, and bismuth sulphide. The proportion of the halogen is determined by the formula $2Cu_2S,Bi_2S_3.2BiSX$, in which X can be replaced by Cl, Br, or I.

The Alkaline Cobaltioxalates.—M. Copaux.—Of the three alkaline cobaltioxalates which are known up to the present time only one has had a definite formula attributed to it:—$Co_2(C_2O_4)_6(NH_4)_6.6H_2O$. The ammonium, potassium, and rubidium salts of this series are isomorphous amongst themselves. The iron, aluminium, and chromium salts differ in form and in composition. This fact, singular in itself, shows that cobalt possesses a specific property. If the double salt of the sesquioxides (of which a table is given in this paper) are specially examined, it is seen that cobalt takes up a position further from iron and nearer to chromium and aluminium.

Constitution of Ammoniacal Cupric Salts. Action of Ammonia.—M. Bouzat.—The author examines the ammoniacal compounds of copper with a view to establishing their composition, and his experiments show that the following law is true: the quantity of heat evolved during the combination of ammonia with a cupric salt derived from a strong acid is the same whichever cupric salt is used. This law can be extended to zinc and all the metals which give with ammonia compounds analogous to ammoniacal copper salts. This law shows that the ammoniacal copper compounds ought to be considered as salts of complex bases.

***p*-*p*-Dinitrohydrazobenzene.**—P. Freundler and L. Béranger.—The nitration of diacetylhydrazobenzene gives as principal product a nitrated derivative, $NO_2.C_6H_4.N(CO.CH_3)N(CO.CH_3)C_6H_4.NO_2$, which crystallises from acetone in yellowish plates, fusing at 186—187°. These are very slightly soluble in alcohol and ether. At the same time a small quantity of *p*-*p*-dinitroazobenzene is obtained, fusing at 220°. The authors examine the constitution of this di-nitrated derivative by transforming it into dinitrohydrazobenzene by means of saponification, and they find it to possess the formula $HO.ON=C_6H_4=N—N=C_6H_4=NO.OH$.

Thiosulpho-carbamic Ether Derivatives of Primary Amines.—Marcel Delépine.—Dithiourethanes of general formula $R.NH.CS.SR'$ Hofmann has shown can be obtained by condensing the sulpho-carbimides with the mercaptans $R.N:C:S+H.SR'=R.NH.CS.SR'$. This reaction has the inconvenience of necessitating the preparation of two constituents with a very unpleasant smell. The author finds that the dithiourethanes of the above type can be easily obtained by the action of a single molecule of a halogen ether on the sulphocarbonic compounds of primary amines; that is to say, by stopping the reaction at the first phase of the preparation of the imido-dithiocarbonic ethers, the reaction taking place according to the equation $NHR.CS.S.NH_3R+XR'=RNH.CS.SR'+X.NH_3R$.

Synthesis of Aldehydes of the Fatty Series by means of Nitromethane.—L. Bouveault and A. Wahl.—The authors succeed in transforming a fatty aldehyde into its superior homologue, in the same manner as in the aromatic series, owing to the action of nitromethane. In the first case they employ isovaleric aldehyde and œnanthol.

Mechanism of Chemical Reactions taking place in Plants submitted to the Influence of Sodium Nitrate.—E. Charabot and A. Hébert.—In the same way as sodium chloride, sodium nitrate has the effect of causing etherification in plants and accentuating the diminution of the percentage of water. These two salts modify in an analogous manner the series of chemical phenomena in plants. The mechanism of their intervention, however, does not appear to be the same.

Composition and Volumetric Estimation of Sodium Methylarsiniate.—MM. Adrian and Trillat.—To determine the composition of sodium methylarsiniate or arrhenal, the authors successively estimate—(1) The quantity of pyroarseniate of sodium obtained by heating the substance with nitric acid and calcining the residue at a dull red heat; (2) the quantity of arsenic in the state of magnesium pyroarseniate; (3) the quantity of water of crystallisation. The true formula is thus proved to be $CH_3AsO(NaO)_2+6H_2O$. The volumetric estimation of this substance is based on the precipitation of sodium methylarsiniate by a known solution of silver nitrate in excess; this excess being afterwards titrated, after filtration, against ammonium sulphocyanate.

MISCELLANEOUS.

Estimation of Copper in the State of Oxalate, and on a Method of Separating Copper from Cadmium, Arsenic, and Tin.—C. A. Peters.—In a solution of sulphate of copper of about 50 c.c. and containing not less than 0·0128 grm. of CuO, the copper can be completely precipitated in the form of oxalate by the addition of oxalic acid, especially in the presence of a moderate quantity of nitric acid (such as 5 c.c. of the concentrated acid). Under these conditions cadmium, arsenic, and iron, and even small quantities of tin (but not zinc) remain dissolved.—*Zeit. Anorg. Chem.*, vol. xxvi., p. 111.

Action of Zinc Powder on the Dibromides, $C_nH_{2n}Br_2$.—V. Ipatief.—Fresh experiments have shown that in the action of zinc powder and alcohol on the bromide, $(CH_3)_2CBr—CH_2—CH_2Br$, the carbide which is formed in the greatest quantity (about 30 per cent), is isopropylethylene, boiling at 21—22°. There is also formed a substance which distils over between wide limits, 80—130°, and contains about 8 per cent of bromine; this is a mixture of non-saturated ether, non-saturated bromide, and perhaps some oxide of dimethyltrimethyleneglycol. According to this, in the formation of the carbide by means of the bromide of α-dimethyltrimethylene, zinc powder, and alcohol, one molecule of HBr is removed at the expense of the Br united to the most hydrogenised C, and the Br united to the tertiary C is replaced by H.—*Journ. Soc. Phys. Chim. R.*, vol. xxxii., p. 647.

The Thiocyanates of Copper and Silver in Gravimetric Analysis.—E. G. Van Name.—The estimation of copper in the state of cuprous thiocyanate has already been recommended by Rivot; the author has modified the method so far as concerns the collection of the precipitate. The cupric solution is first treated with bisulphite of sodium, then with an excess of sulphocyanate of ammonium. The precipitate of $(CNS)_2Cu_2$ is collected on a layer of asbestos in a pierced crucible of which the weight is known; it is then washed with cold water, dried at 110°, and weighed. The estimation of the silver is effected in almost the same manner, except that no bisulphite is used. The $CNS.NH_4$ should be free from chloride; and further, should not be used in excess, as it re-dissolves the precipitate.—*Zeit. Anorg. Chem.*, vol. xxvi., p. 230.

ERRATA.—The Papers by Mr. R. S. Hutton, B.Sc. (p. 159), and Mr. R. A. Taylor, F.C.S. (p. 269), were reprinted from the *Memoirs and Proceedings of the Manchester Literary and Philosophical Society.*

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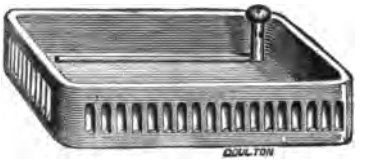

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Manuals: E. Chr. Hansen, "Practical Studies in Fermentation," London (Spon), 1896). Alfred Jörgensen, "Micro-Organisms and Fermentation," London and New York (Macmillan and Co.), 1900.

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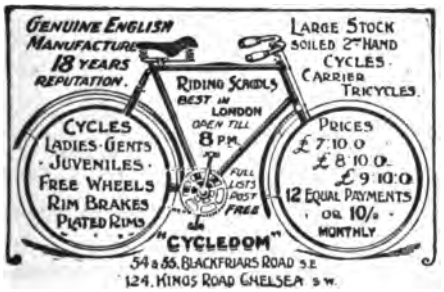
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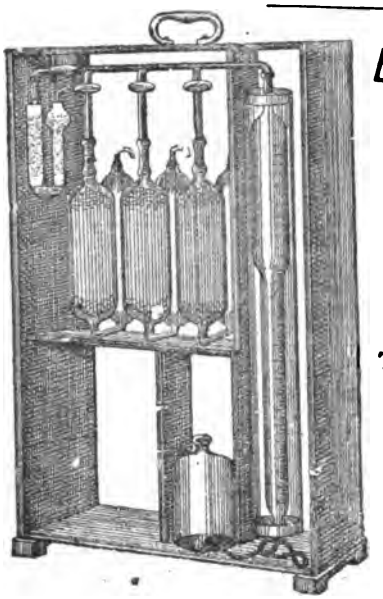

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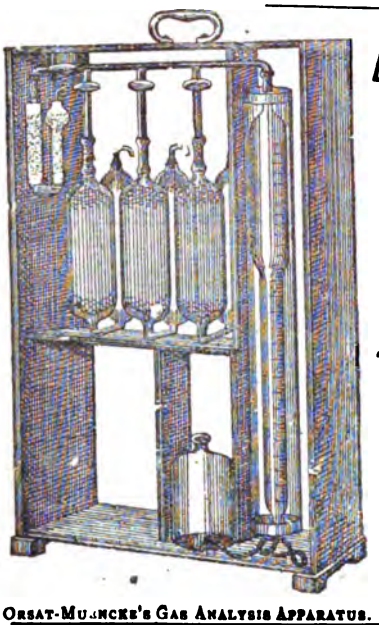

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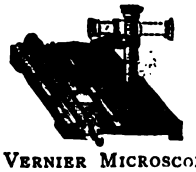

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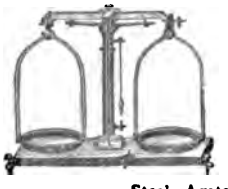

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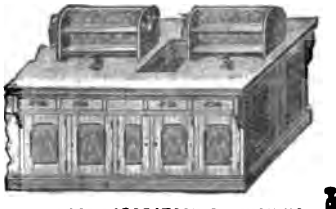

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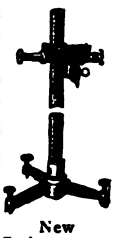

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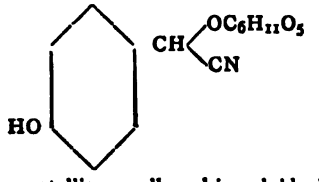
CYANOGENESIS IN PLANTS.*

Part II.—The Great Millet, *Sorghum vulgare*.†

By WYNDHAM R. DUNSTAN, M.A., F.R.S.,
Director of the Scientific Department of the Imperial Institute,
and
T. A. HENRY, D.Sc., Lond.

The authors have investigated the nature of the poison contained in the young plants of *Sorghum vulgare*, the Great Millet or Guinea Corn (the *Jwár* of India or *Dhurra shirshabi* of Egypt). This plant is cultivated in tropical countries for the sake of the seed, which is important as a food grain. The young plants have proved fatal to animals, especially in Egypt, where the attention of the authors was directed to the subject by Mr. E. A. Floyer, of Cairo, who has kindly provided the material required for the investigation.

The authors show that the young plant, but not the seeds or old plants, when crushed with water furnishes prussic acid (about 0·2 per cent of the dried plant). The acid is not present in the free state, nor is it produced by acting on the plant with boiling water or with alcohol. The production of the poison is due to the action of a hydrolytic enzyme, apparently identical with the emulsin of bitter almonds on a cyanogenetic glucoside which has been named "dhurrin," from the Arabic name for the plant, "dhurra." This glucoside has been proved to be derived from parahydroxymandelic nitrile by the association of the residue of one molecule of dextrose. Its formula is therefore $C_{14}H_{17}O_7$,—



Dhurrin crystallises well, and is soluble in both water and alcohol. When hydrolysed by emulsin or by dilute acids it is converted into parahydroxybenzaldehyde, dextrose, and hydrocyanic acid according to the equation $C_{14}H_{17}O_7N + H_2O = C_7H_6O_2 + C_6H_{12}O_6 + HCN$. When warmed with alkalis, dhurrin is resolved first into dhurrinic acid and ammonia. This acid subsequently undergoes further hydrolysis when warmed with dilute hydrochloric acid into parahydroxymandelic acid and dextrose (1) $C_{14}H_{17}O_7N + H_2O = C_{14}H_{18}O_9 + NH_3$, (2) $C_{14}H_{18}O_9 + H_2O = C_8H_8O_4 + C_6H_{12}O_6$.

The identity of the parahydroxymandelic acid was established by its synthesis from the cyanhydrin of parahydroxybenzaldehyde.

Dhurrin differs from the other two known cyanogenetic glucosides, the amygdalin of bitter almonds and the lotusin found by the authors in *Lotus arabicus*, in being derived from dextrose and not from maltose.

The authors point out the protective purpose served by the existence of the cyanogenetic glucoside in the young plant.

The authors intend to fully investigate the several problems which are raised by the occurrence of cyanogenetic glucosides in plants.

They are at present engaged in examining several other plants which have furnished prussic acid, among them being *Manihot utilissima*, *Linum usitatissimum*, *Lotus australis*, and *Phaseolus lunatus*.

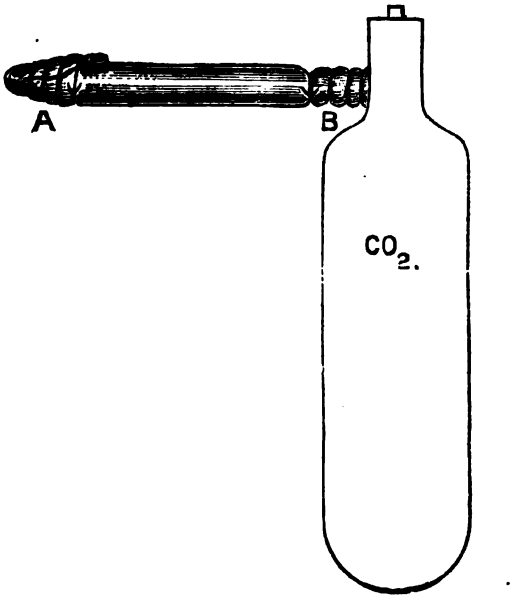
* Abstract of a Paper read before the Royal Society, May 15, 1902.

† The authors' previous paper, entitled "The Nature and Origin of the Poison of *Lotus arabicus*" (*Proc.*, 1900, vol. lxvii., p. 224; 1901, vol. lxviii., p. 374; and *Phil. Trans.*, B., 1901, excv., p. 515), is to be regarded as Part I. of this series.

A SIMPLE METHOD OF COLLECTING SOLID CARBON DIOXIDE FOR LECTURE PURPOSES.

By CHAS. R. DARLING, A.R.C.Sc.(Ire.), A.I.C., F.C.S.

Most lecturers must have experienced disappointment in attempting to collect solid carbon dioxide in quantity by means of the metallic box sold for the purpose. This arrangement is inefficient for two reasons; firstly, being made of thin metal and possessing a large surface, it affords every facility for the passage of heat from the atmosphere to the interior of the box; and secondly, the meshes placed in the handles are not sufficiently close to retain the particles of solid. Far better results are obtained by the method of tying a piece of flannel round the nozzle of the cylinder, although in this case also a large quantity of the solid escapes through the single layer of flannel.



The following method, which to the best of my knowledge has not previously been published, will be found to give excellent results:—A piece of coarse flannel (such as is used for floor-cloths), about 15 inches square, is rolled round a piece of rod or tubing 1½ inches or more in diameter.

The rod is partially withdrawn, and one end of the flannel bent over on itself, and tied with string, as shown at A. The rod is then completely removed, and the other end of the flannel tube tied with string round the nozzle of the CO_2 cylinder, as at B. The gas is then turned on for a short time until the whole length of the flannel feels hard to the touch. On cutting the strings, and removing and unrolling the flannel, a solid rod of carbon dioxide, much denser and harder than that obtained by the usual methods, and about 8 inches long, will be found. This may be kept for a considerable time rolled up in the flannel, or may be transferred to a Dewar vacuum vessel, and used for several experiments.

I have used this method for some time for lecture purposes, and on occasions when a quantity of solid carbon dioxide has been required for cooling purposes, and have found it uniformly successful. There is no restriction as to the diameter of the rod round which the flannel is wound, providing the material is large enough to pass four or five times round the rod. The efficiency of the method is no doubt due to the retention of the finest particles of solid, owing to the several layers of flannel through which the gas must escape, and also to the heat-insulating properties of the material which serve to protect the solid from extraneous heat. The preparation of solid carbon dioxide by this method will be found to form a most striking lecture experiment.

Physical Laboratory,
City and Guilds Technical College, Finsbury, E.C.

CONTRIBUTIONS TO THE CHEMISTRY OF THE RARE EARTHS OF THE YTTRIUM GROUP.*

I.

By L. M. DENNIS and BENTON DALES.

(Concluded from p. 293).

Treatment with Potassium Trinitride, KN_3.

The method proposed by Dennis and Kortright (*Zeit. Anorg. Chem.*, vi., 35) for the separation of thorium from members of the cerium and yttrium groups, by means of potassium trinitride, was tried to see if perhaps some reaction similar to the one for thorium could be found. The results were entirely negative. Partial precipitation by potassium trinitride was found to fraction the rare earths of this group rather slowly. The precipitate in the case of thorium was thorium hydroxide. In this case it seemed rather to be a basic nitrate. It was white, very finely divided, and adhered to the beaker, and had also a tendency to go through the filter.

A dilute solution of the nitrate ($R^{III} = 112{\cdot}16$) was treated with a dilute solution of potassium trinitride containing free hydronitric acid and boiled. A precipitate was obtained on boiling for two or three minutes. It was filtered, washed with hot water, dissolved in nitric acid, and converted into oxalates. The atomic weight was 118·96. The nitric acid solution was pink, and the absorption bands were practically the same as in the original solution.

Fractional Precipitation with Ammonia.

The work of many chemists who have investigated the earths of the yttrium group has shown that these elements can unquestionably be partially separated by a long series of fractions with dilute ammonium hydroxide, but the work is so extremely tedious and the separations are so very slow that it is questionable whether this procedure can be considered to be of material value. As an illustration of the slight effect of fractionation by this reagent one experiment will suffice.

A very dilute solution of the neutral chlorides containing about 57 grms. of the oxides in 12 litres was treated with an amount of dilute ammonia sufficient to precipitate only one-tenth of the earths present. During the addition of the ammonia the solution was vigorously stirred by blowing through it a blast of air, and this stirring was continued for about an hour. A precipitate formed very slowly. The hydroxides were then washed by decantation until the wash-water gave no precipitation with ammonia. The mother-liquor and wash-waters were evaporated to the original volume, and precipitated again with the same amount of ammonia. The atomic weights of the earths precipitated in these two treatments were 103·9 and 104·0, while that of the original material was 96.

Fractionation with Ammonium Carbonate and Dilute Acetic Acid.

Mosander (*Phil. Mag.*, xxiii., 251; *Liebig's Ann. Chem.*, xlviii., 219) in 1843 mentioned that the hydroxides of the rare earths of this group are soluble in a concentrated solution of ammonium carbonate, and that they could be fractioned in this way. Krüss also mentioned this solubility (*Liebig's Ann. Chem.*, cclxv., 1).

This treatment alone was first tried here. The results were much better than was expected. The rare earth hydroxides were precipitated by ammonia (the latter need not be washed out). By a single treatment of these hydroxides with a quantity of a saturated solution of ammonium carbonate sufficient to dissolve one-fourth of the whole, an earth mixture was dissolved whose metal atomic weight was 121·41. The bases in the undissolved portion had an atomic weight of 107·94, while the original solution contained a metal mixture of atomic weight 108·08.

Quantities of saturated ammonium carbonate solution sufficient to dissolve other amounts of the precipitated hydroxides than one-fourth were then tried on different portions of this earth-mixture. An amount of carbonate solution sufficient to dissolve one-tenth of the hydroxides precipitated was added to one portion, while to others were added amounts of the carbonate solution sufficient to dissolve nine-tenths, one-fifth, one-third, and one-half. On the whole, however, the addition of one-fourth or perhaps one-fifth the amount of carbonate solution necessary to dissolve all the hydroxides seems to be most suitable for this method of fractionation. The total precipitation of the earths by a solution of ammonium carbonate with partial solution of the precipitate in an excess of that reagent was also tried, but this is no better than precipitation with ammonia and partial solution in ammonium carbonate. A second extraction of the hydroxides from the undissolved portion ($R^{III} = 107{\cdot}94$) from the first treatment was made, and the atomic weight of the metal mixture which dissolved was 125·91. Evidently the material could be thus extracted at least twice with profit.

To the dissolved portion mentioned above ($R^{III} = 121{\cdot}41$) there was then added very dilute acetic acid (1 : 30) slowly and with constant stirring, first adding concentrated acetic acid just to turbidity. This treatment had a most marked effect. The granular precipitate thrown out was of a pink colour, and when dissolved showed all the absorption bands very strongly, while the bands in the remaining solution were much weakened.

When the dilute acetic acid had been added as above, a point was reached at which the addition of more acid would produce no further precipitate. This is due to the fact that the rare earth hydroxides of this group are nearly as soluble in ammonium acetate as in ammonium carbonate, and a state of equilibrium is soon reached between the three, after which dilute acetic acid has no effect upon the double ammonium salts of the rare earths. It was necessary, when this point was reached, to filter, decompose the ammonium carbonate solution with hydrochloric acid, dissolve the precipitate which was formed by further addition of the acid, and then to precipitate the whole with ammonia, wash, re-dissolve in hydrochloric acid, and re-precipitate with ammonia. The hydroxides were then dissolved in saturated ammonium carbonate solution, and the precipitation by dilute acetic acid repeated.

This particular material after having been treated three times with ammonium carbonate and dilute acetic acid in the manner above described, gave a very small amount (less than 1 grm.) of an earth which yielded a white oxalate and a white oxide. A determination, not in duplicate, of its metal atomic weight gave the interesting result of 170. In the dry oxide only, the green absorption band of erbium was visible by reflected light. In saturated solution this earth mixture did not show the absorption

* *Journal of the American Chemical Society*, vol. xxiv., No. 5.

bands of holmium, thulium, nor dysprosium, but those of erbium and samarium were visible. They were, however, much weaker than in the original solution. The spark spectrum of this solution was also examined, and the following lines of ytterbium as given by Thalén were found (*Journ. de Phys.*, [2], ii., 37) :—

Angle.	Wave-length.	Thalén's measurements.
10° 57' 00"	6220·9	6221·0 (1)
9° 46' 00"	5555·6	5555·5 (1)
9° 37' 30"	5475·7	5476·0 (1)
9° 24' 30"	5353·5	5352·0 (1)
9° 22' 30"	5334·8	5334·0 (1)
9° 18' 50"	5300·3	5300·0 (4)
8° 24' 10"	4785·7	4785·5 (2)
8° 17' 40"	4724·5	4725·0 (2)

The spark spectra of erbium and samarium were very weak. None of the characteristic gadolinium lines were found. The mixture was, therefore, ytterbium and erbium with some samarium, but no holmium, thulium, or dysprosium, no yttrium, gadolinium, or terbium.

To show further the effectiveness of this method of fractionation, it was tried on nearly all of the material in hand. The material was divided into two parts, in one of which the bases had an atomic weight of 117·37, and in the other of 107·15. Each of these portions was carefully purified by treating the boiling, slightly acid chloride solution with hydrogen sulphide, filtering, boiling to expel hydrogen sulphide, then precipitating with ammonia and washing, dissolving the precipitate in dilute hydrochloric acid, neutralising, and precipitating with oxalic acid solution, stirring the while by blowing air through the solution. The oxalate precipitate was washed with 0·1 per cent hydrochloric acid to remove iron, then with water, dried, ignited, and dissolved in hydrochloric acid. The atomic weights given above were obtained from the material thus purified by precipitating this chloride solution again with oxalic acid, both solutions being boiling hot.

The details of the treatment for the solution (R^{III} = 117·37) were as follows :—The chloride solution was divided and placed in three tall cylinders, diluted and precipitated with ammonia. The hydroxides were washed, then dissolved completely in saturated ammonium carbonate solution, and concentrated acetic acid added just to turbidity. Then to each of the three cylinders was added 1000 c.c. of dilute acetic acid (1 part acid diluted to 30 with water) in small quantities at a time, and with constant stirring by a current of air. The flocculent precipitate soon became granular, and could easily be separated from the mother-liquor by decantation. The last of the mother-liquor was removed from this fraction 1 by suction. Dilution with water caused the earths to precipitate from the ammonium carbonate solution; therefore the mother-liquor could not be washed out. The mother-liquors were treated with concentrated hydrochloric acid till the ammonium carbonate was destroyed, and the resulting hydroxides dissolved, then precipitated with ammonia, dissolved in hydrochloric acid, and re-precipitated with ammonia; then these hydroxides were treated again with saturated ammonium carbonate solution. This time the carbonate solution was placed in two cylinders, the excess of ammonium carbonate neutralised by concentrated acetic acid, and 700 c.c. of dilute acetic acid added to each one slowly, and with constant stirring. The precipitate was fraction 2.

The mother-liquors here were removed and decomposed exactly as before, and the ammonium carbonate solution of the hydroxides neutralised with concentrated acetic acid, and treated in one cylinder with about 600 c.c. of dilute acetic acid (fraction 3). The mother-liquor from this fraction still showing a trace of the erbium band in the green after its conversion by the usual decomposition into chloride solution and concentration, it was diluted, precipitated with ammonia, dissolved in saturated ammonium carbonate solution as before, and precipitated this time after neutralisation by about 30 c.c. of dilute acetic acid. The precipitate (fraction 4) was, of course, slight. The mother-liquor from it was dissolved in hydrochloric acid, nearly neutralised with ammonia, and precipitated with oxalic acid to remove most of the iron. The oxalates were washed, dried, ignited, and dissolved in hydrochloric acid. This solution concentrated to saturation gave three absorption bands, the red and the green of erbium, and the samarium band between the blue and the green, none of them strongly. The atomic weights of the bases for the series were :—

Original, 117·37.

1.	2.	3.	4.	Residual mother-liquor.
115·25	116·33	141·62	159·78	165·48

The other material (R^{III} = 107·15) treated in the same manner yielded the following series of atomic weights :—

1.	2.	3.	4.	Residual mother-liquor.
105·33	102·4	110·6	119·45	146·53

It is worthy of note here that the metal atomic weights of the first two of the fractions in each of these series are quite close together, while those of the other fractions show a marked divergence from one another. In the second series the curve of atomic weights even goes through a minimum. There are two reasons for the apparent non-fractionation in these first two fractions. One is that the larger portion of the material used was precipitated in these first two fractions. The other and more important one is that the erbium (R^{III} = 166), terbium (R^{III} = 159), and yttrium (R^{III} = 89) have so distributed themselves in the first and second fractions, more terbium in the first, more erbium in the second, that the atomic weights of the metal mixtures are about the same.

The changes in colour of the ignited oxides from these series were very marked. In the first series the original earth was a deep orange-yellow colour. No. 1 gave a more deeply coloured earth, No. 2 was chamois-coloured, No. 3 pink, No. 4 white with a pinkish cast, and the earth from the mother-liquor almost white. In the second series the earth from the original solution had a deeper orange-yellow colour than that from the other original solution. No. 1 was more deeply coloured yet than this original solution, No. 2 lighter, No. 3 chamois-coloured, No. 4 the same, and that from the mother-liquor white with a pinkish cast.

This colour change evidences a marked change in the terbium content of the various fractions. Terbium concentrates very markedly in the first fractions. It is true that much the larger portion of the earths was thrown out in the first fraction in these two series, but by a short systematic fractionation, erbium material can unquestionably be obtained terbium-free.

This method seems then to offer a method for the comparatively rapid concentration of terbium with yttrium at one end of the series, and of erbium and ytterbium at the other. Holmium, thulium, and dysprosium concentrate in the middle fractions.

The results thus far obtained in this investigation may be briefly summarised as follows :—

The Gibbs method of determining the equivalent weight does not give exact results even when the conditions prevailing in different series of determinations are identical.

There is a slight error in the sulphate method of determining the equivalent weights, due to the formation of some acid sulphate.

Of the many methods of partial precipitation which were investigated the following seem to be the most rapid ; primary potassium oxalate, potassium trinitride, partial decomposition by fusion of the nitrates with alkali nitrates, and electrolysis of neutral solutions. Magnesia usta causes fractionation of this material, but the progress of

the separation can not be easily controlled with the spectroscope as was done by Muthmann in his work on didymium.

One of the best methods for the separation of yttria from the other members of this group is that of fractional precipitation of a neutral solution by potassium chromate. Quite pure yttria may be obtained in this manner at the end of a comparatively short series of fractions.

Unusually rapid separation of the earths of this group is effected by ammonium carbonate and acetic acid. Fractional solution of the hydroxides by means of a saturated ammonium carbonate solution causes quite rapid separation, and if this ammonium carbonate solution be fractionally precipitated by addition of acetic acid, the results are most striking. Ytterbium is the last of the earths to be precipitated by this treatment. Erbium and terbium concentrate in the first fraction.

THE ADIO-ACTIVITY OF THORIUM COMPOUNDS.*

I. An Investigation of the Radio-active Emanation.

By E. RUTHERFORD, M.A., D.Sc.,
Macdonald Professor of Physics, McGill University, Montreal,
and
FREDERICK SODDY, B.A. (Oxon.),
Demonstrator in Chemistry, McGill University, Montreal

(Concluded from p. 295).

Effect of Conditions upon Emanating Power.

Before any further work was undertaken, it was necessary to make a close study of the influence of conditions upon the emanating power of thorium compounds.

Effect of Temperature.—The effect of increase of temperature on the emanating power of thoria has already been fully investigated by one of us (*Phys. Zeit.*, 1901, ii., 429). The results, stated briefly, show that an increase in temperature up to a certain limit, in the neighbourhood of a red heat, correspondingly increases the emanating power. At the maximum, this is between three and four times the value at the ordinary temperature, and is maintained at this increased value for several hours without any sign of diminution with time. When the thoria is allowed to cool, the emanating power then returns to the neighbourhood of the normal value. If, however, the limit of temperature given is exceeded, de-emanation sets in, and even while the high temperature is maintained, the emanating power falls rapidly to a fraction of its former value. On cooling, the substance is found to be more or less de-emanated. It is of interest that no increase of emanating power is observed when de-emanation commences.

These experiments were extended to include the effects of cooling. The platinum tube which contained the thoria was surrounded with a felt jacket containing a mixture of solid carbon dioxide and ether. The emanating power immediately fell to 10 per cent of its former value. On removing the cooling agent, it again rose quickly to nearly its normal value.

In another experiment, some thoria was surrounded in a platinum crucible with a mixture of solid carbon dioxide and ether, and kept in a vacuum for several hours. On removing it and allowing its temperature to rise, it possessed much the same value as an ordinary sample, and after standing some time in the air it was again tested and no difference could be detected between the two.

Thus changes in temperature produce very marked simultaneous changes in emanating power, but between the limits of −110° and an incipient red heat no permanent alteration in the value occurs.

Effect of Moisture.—Dorn (*loc. cit.*) had noticed that moisture produced a moderate increase in the power of thoria of giving an emanation, and of exciting radio-activity on surrounding surfaces. We have confirmed and extended his results by the following experiments:—

Two similar weights of ordinary thoria were exposed in jars sealed with wax, the one containing sulphuric acid and the other water, for a period of four days. The desiccated sample showed 54 per cent, and the sample exposed to water-vapour 134 per cent of the original emanating power. The experiment was repeated and the samples left for a week with much the same result: 70 per cent and 141 per cent respectively. It was of interest to see if a more complete desiccation would further reduce the emanating power. Five grms. of thoria were sealed up in a tube containing phosphoric oxide, the two substances being separated by a plug of glass-wool. Before sealing, the tube was exhausted by a Töpler mercury pump. After twenty-six days the end of the tube was connected with a closely packed phosphoric oxide tube, the tip broken off inside the connection, and a slow stream of dried air thus allowed to enter. The other end was connected to the testing cylinder, and arrangements were made to send a stream of air through into the cylinder. When all was ready, this end of the tube was broken inside the connection, and the emanating power measured. A similar experiment made with an ordinary sample of thoria, using the same arrangement, showed that the desiccated sample possessed 79 per cent of the emanating power of the ordinary sample tested under the same conditions.

A sample of thoria sprayed with water gave 125 per cent of its original emanating power. If completely flooded with water, however, the value is much reduced, as would be expected from the reduction of surface.

Another trial was made, in which thoria was flooded with concentrated sulphuric acid. Hardly any emanation was observed so long as the mixture remained undisturbed, but when vigorously shaken it gave nearly one-half of the original emanation.

These experiments show that the presence of water, although producing a marked increase, is not apparently essential for the production of the phenomena. It must be mentioned, however, that thoria only ceases to lose weight after prolonged ignition with the blowpipe; that is, under conditions which nearly destroy its emanating power. This, with analogous points, will be taken up however, in a separate communication on the more purely chemical side of the question.

The results of some experiments on the effects of other conditions may be shortly tabulated. In each case the sample was exposed to the conditions given for four days. The emanating power is that possessed at the end of this period, compared with that of the first sample, which is regarded as 100 per cent:—

1. Kept in sealed test-tube enclosed completely in lead tube	100 per cent
2. Taken from tightly stoppered stock bottle containing the main quantity	100 „
3. Sealed up in test-tube and exposed to bright all-day sun	100 „
4. Exposed to the air of the laboratory in open watch-glass	105 „
5. Kept in a continuous stream of ordinary air	88 „

The last experiment was made at a different time from the other four, and therefore is not strictly comparable. The most useful result attained is that thoria does not change in emanating power when kept in closed vessels under different conditions, but when exposed to the air the emanating power varies within comparatively narrow limits.

Thorium Hydroxide.—The effect of time on the emanating power of the freshly prepared hydroxide already mentioned is one of the most striking observations in this connection. The following additional experiments have been made on this point. A quantity of hydroxide was prepared, and separate portions subjected to different

* Communicated by the Authors.

drying temperatures and subsequent conditions, as follows :—

	Emanating power.
1. Dried at 110° and exposed some hours to the air	264 per cent
2. Dried as before at 110° and kept in desiccator until tested	226 ,,
3. Dried at 200° and kept in desiccator..	220 ,,
4. Dried at 250° and kept in desiccator..	219 ,,

From this, it appears that the additional loss of water caused by exposure to increasing temperatures is without effect on the emanating power.

A similar experiment to that described for thorium oxide was performed with the hydroxide. Two quantities were exposed in closed bottles to the action of moist air and of air dried with sulphuric acid respectively, and showed, after four days, emanating powers of 394 per cent and 307 per cent. After having been exposed to the air for twenty-four hours, these samples showed 350 per cent and 324 per cent respectively.

The next experiment was designed to include the effect of carbon dioxide, which the hydroxide absorbs from the air to the extent of 2 per cent of its weight. A quantity of hydroxide was tested immediately after preparation, and possessed 140 per cent emanating power. A sample was sealed up in a test-tube, while another similar sample was tested in the following manner. It was exposed to a current of moist carbon dioxide for an hour, and then possessed an emanating power of 156 per cent. It was then left exposed to the air of the laboratory and tested at intervals :—

After two days	Emanating power	263 per cent
After six days..	,,	325 ,,
After ten days	,,	300 ,,
After eleven days	,,	341 ,,
After sixteen days.. ..	,,	362 ,,

On the last day, the sealed-up specimen was opened and examined, and was found to possess an emanating power of 298 per cent. These experiments show that if the air is fundamental in producing the increase of emanating power with time, a very limited quantity of it is effective. For the present, it is perhaps better to consider it as an effect of time simply, hastened no doubt by the presence of water-vapour.

On the Chemical Nature of the Emanation.

The following work has reference to the emanation itself, and not to the material producing it, and was designed to see whether the emanation possesses chemical properties which would identify it with any known kind of matter. It had been noticed at the time of its discovery that it passed unchanged through concentrated sulphuric acid. The same holds true of every reagent that has been investigated.

The effect of temperature was first tried. The air containing the emanation, obtained in the usual way by passage over thoria, was led through the platinum tube heated electrically to the highest attainable temperature, and also through the tube cooled by solid carbon dioxide and ether. The tube was then filled with platinum black, and the emanation passed through in the cold, and with gradually increasing temperatures, until the limit was reached. The effect of the intense heat was to convert the platinum black completely into platinum sponge. In another experiment, the emanation was passed through a layer of red-hot lead chromate in a glass tube. The current of air was replaced by a current of hydrogen and the emanation sent through red-hot magnesium powder and red-hot palladium black, and, by using a current of carbon dioxide, through red-hot zinc dust. In every case, the emanation passed without sensible change in the amount. If anything, a slight increase occurred, owing to the time taken for the gas current to pass through the tubes when hot being slightly less than when cold, the decay *en route* being consequently less. It will be noticed that the only known gases capable of passing in unchanged amount through all the reagents employed are the recently discovered gases of the argon family.

But another interpretation may be put upon the results. If the emanation were the manifestation of excited radio-activity on the surrounding atmosphere, then since from the nature of the experiments it was necessary to employ in each case, as the atmosphere, a gas not acted on by the reagent employed, the result obtained might be explained. Red-hot magnesium would not retain an emanation consisting of radio-active hydrogen, or red-hot zinc dust an emanation consisting of radio-active carbon dioxide. The correctness of this explanation was tested in the following way :—Carbon dioxide was passed over thoria, then through a T-tube, where a current of air met and mixed with it, both passing on to the testing cylinder. But between this and the T-tube, a large soda-lime tube was introduced, and the current of gas thus freed from its admixed carbon dioxide before being tested in the cylinder for emanation. The amount of emanation found was quite unchanged, whether carbon dioxide was sent over thoria in the manner described, or whether an equally rapid current of air was substituted for it, keeping the other arrangements as before. The theory that the emanation may consist of the surrounding medium rendered radio-active is thus excluded, and the interpretation of the experiments must be that the emanation is a chemically inert gas analogous in nature to the members of the argon family.

It is perhaps early to discuss these results from a theoretical point of view, although it appears certain that an explanation of the nature of the emanation must precede, as a necessary step, any hypothesis put forward to account for emanating power. The explanation already advanced and disproved being left out of the question, two other views of the origin and nature of the emanation are still possible. It may be that one of the inert constituents of the atmosphere is rendered radio-active in the presence of thoria, and so constitutes the emanation. The actual amount being probably extremely small, and air being a constant impurity in all gases as ordinarily prepared, it is of course no argument against this view that emanating power is independent of the gaseous medium surrounding the emanating material. An experiment is in progress, however, to ascertain whether emanating power persists in a current of gas as free from air as present methods of preparation allow. The other alternative is to look upon the emanation as consisting of a gas emitted by the thorium compound. It is not necessary that such should contain thorium, it might conceivably be an inert gas continuously emitted in the radio-active state.

In the present state of knowledge, it would be premature to attempt to choose between these two alternatives. But in any decision of this point, the work already given on the regeneration of the emanating power of thoria de-emanated by ignition, the continuous loss of emanating power by successive ignition at increasing temperatures, and the increase in the chemical activity of thorium hydroxide with time, must be taken into consideration.

Concentration of the Radio-active Material.

Since the preceding account was written, developments have been made in the subject which completely alter the aspect of the whole question of emanating power and radio-activity. The first has reference to thorium nitrate, which in the solid state hardly possesses any emanating power. In a careful determination, using 20 grms. of the finely-powdered commercial salt, this worked out to be only 1·8 per cent of the emanating power of thoria. Dissolved in water, however, and tested for emanation by bubbling a current of air through it, it gives about three times as much emanation as thorium oxide; that is, solution in water increases the emanating power of thorium nitrate nearly two hundred times. The emanating power, as in the case of solids, is proportional to the weight of

substance present, and within the limits tried is not much affected by dilution, for a solution of 10 grms. made up to 25 c.c. in volume possessed a similar value when diluted four times.

Solutions of thorium chloride also give a large amount of emanation.

In these experiments, the cylinder c (Fig. 1) is replaced by a Drechsel bottle. A drying tube of calcium chloride is inserted between it and the testing cylinder to prevent the moisture destroying the insulation of the latter. In this connection, the method of testing the insulation by varying the voltage is invaluable. The air current under these circumstances cannot of course be kept so constant as when working with solid substances, and the results are not strictly comparable in consequence, but the arrangement works well enough for a first approximation.

Simultaneously with this observation of the latent emanating power of thorium nitrate, it was noticed that preparations of thorium carbonate varied enormously in emanating power according to their method of preparation. A sample prepared from the nitrate by complete precipitation with sodium carbonate showed an emanating power of 370 per cent of that of the ordinary oxide, and this value remained fairly constant with time. In another experiment, the precipitated carbonate was partially re-dissolved in nitric acid, and the re-dissolved fraction completely re-precipitated with ammonia as hydroxide. The result was remarkable; the carbonate had an emanating power of only 6 per cent, the hydroxide one of 1225 per cent of that of the ordinary oxide. On repeating the experiments, both fractions proved almost equally inactive, the carbonate showing 14 per cent and the hydroxide 19 per cent of the emanating power of thoria. An even greater difference between these two similar experiments was observed in the effects of time on the different preparations. In the first, the carbonate did not alter in value in seven days, whilst the hydroxide steadily decreased:—

	Hydroxide. Per cent.	Carbonate. Per cent.
Original	1225	6·2
After one day	1094	8·4
After four days..	696	4·8
After seven days	614	4·7
After fourteen days.. ..	473	—

In the second experiment, the emanating power of both the carbonate and hydroxide had increased many fold when tested eleven days later, and the former now possessed 109 per cent, the latter 273 per cent (originally 14 per cent and 19 per cent respectively).

The straight-line radio-activity of the carbonate from the first experiment which possessed such a low emanating power is of interest. It proved to be similar to that of a specimen of hydroxide of normal emanating power, which it resembled in density and state of division. After having been kept seven days without showing any sign of recovering its emanating power, it was re-dissolved in nitric acid, and re-precipitated with ammonia as hydroxide. The latter now possessed, when first made, an emanating power of 65 per cent, and after twenty-four hours 145 per cent, from which value it did not much alter.

These results throw a new light on the question of emanating power. In the first experiment, which we have so far not succeeded in repeating, by an accident in the conditions apparently, two fractions were separated from thorium which varied in their emanating power in the ratio of 200 to 1. The active fraction diminished to nearly a third of its original value in fourteen days spontaneously, whilst the activity of the inactive fraction was, to a large extent, regenerated by solution and re-precipitation, in an exactly analogous manner to the behaviour of thoria de-emanated by ignition. Attempts to repeat this result have so far led to the production of two more or less completely de-emanated fractions, which, however, spontaneously increase in activity with time, as in the second experiment, and this seems to be generally the case, whether incomplete precipitation is effected as in the experiment given by re-solution of the carbonate in acid, or by using a deficiency of sodium carbonate in the first instance.

The production of preparations of such low emanating power led naturally to an examination being made of the filtrates and washings for radio-activity. It was found that these possess when concentrated both emanating power and radio-activity in considerable amounts, although from the nature of their production they should be chemically free from thorium. The behaviour is quite general, a dilute solution of thorium nitrate, after the thorium has been precipitated as hydroxide with ammonia, shows, when concentrated, an emanating power of from one-third to two-thirds that of the original nitrate in solution. It does not matter whether the thorium is precipitated with ammonia directly, or after preliminary partial precipitation as carbonate—either by adding insufficient sodium carbonate in the first place, or by precipitating completely and dissolving part of the precipitate in nitric acid—the thorium-free filtrate invariably possessed emanating power, and when evaporated to dryness exhibited straight line radio-activity also in amounts very much greater than possessed by the same weight of thoria.

The result of a careful chemical investigation of the active filtrates produced under the various conditions described, was to show that these contained no thorium, or at most only a minute trace, but another substance in very appreciable quantities which can be precipitated with sodium phosphate, and which so prepared is a white substance possessing both emanating power and radio-activity, often many hundredfold greater than thoria. It has not yet been obtained in sufficiently large quantities for an exhaustive chemical investigation, and it is impossible at present to say what it may prove to be.

We may at once state, however, that we do not incline to the view that it is ThX either in the sense of the radio-active or emanating constituent of thorium. The evidence of a long series of experiments in two directions, of which the final steps can only find place here, is quite definite on this point, and in our opinion admits of only one conclusion. There seems little doubt of the actual existence of a constituent ThX to which the properties of radio-activity and emanating power of thorium must be ascribed, but in all probability it is present in altogether minute amount, and must therefore be possessed of these qualities to a correspondingly intense degree.

But before the reasons for this view are put forward, it is necessary to discuss more nearly the meaning of the experiments already given on the emanating power. It has been shown that this is a most uncertain quantity, similar experiments often giving preparations of very varying value, as is clearly shown in the results given, as well as in many others in the same direction. The most pregnant fact is, that although, as has been shown, precipitation with ammonia invariably leaves behind considerable emanating material in the filtrate which is lost, this seems to exert little influence on the emanating power of the precipitates. These prepared under different conditions, often by a different number of precipitations, in which therefore varying amounts of the emanating material are lost, show a surprising uniformity in this property, especially after they have attained their maximum power by keeping. It is only necessary to quote the experiment on the almost completely de-emanated carbonate, which gained in emanating power thirty times by conversion into the hydroxide, although during the process much emanating material must have been lost, to show that the value of the emanating power alone furnishes no criterion of the amount of emanating material present.

It may safely be said that three things must be carefully distinguished between in considering the nature of the property possessed by thoria of giving out a radio-active

emanation. First, the nature of the emanation itself; secondly, the nature of the emanating power; and thirdly, the nature of the emanating material. The first, the emanation itself, we have shown to possess the negative properties of a chemically inert gas, whose radio-activity is unaffected by any conditions apparently except lapse of time. With regard to the second—the emanating power, or *rate* at which the emanation is produced per unit weight of substance—it is certain that this does not depend only or even mainly on the quantity of emanating material present. The regeneration of the emanating power of thoria de-emanated by ignition, the enormous variation with time in the emanating power of the hydroxide and carbonate under certain conditions, and the comparatively constant maximum which these substances ultimately attain, although prepared under conditions where different amounts of the emanating material are lost, make this point perfectly clear. These considerations, taken in conjunction with the effect of temperature, moisture, &c., on emanating power, and the nature of the emanation itself, make the property appear rather as the result of a dynamical change, possibly in the nature of a chemical reaction where the active mass of emanating material is a constant—than as the property of a peculiar kind of matter in the static state, additive with regard to mass.

It is, however, neither the emanation itself nor the emanating power with which we are concerned in these experiments, but the third conception—the emanating material, *i.e.*, the substance, whether thorium or not, which is responsible for the activity. It has been shown that it is difficult to follow by means of the value of the emanating power, the progress of the removal of the active material. When this was realised, attention was directed to the straight line radio-activity, which is generally unaffected by these changes of conditions and previous history which produce such profound alteration in the former property. The two phenomena are undoubtedly connected. The intensely radio-active preparations obtained from thorium in different ways always show correspondingly great emanating power, when the conditions are favourable for the manifestation of the latter. Solution appears to be the most generally favourable condition. The experiments we had been engaged in were therefore repeated in a form which would allow a close study of the total radio-activity, in the hope that this value would prove a more suitable indication of the amount of active material present than the emanating power alone.

Seventy grms. of thorium nitrate were dissolved in 4 litres of boiling water, and precipitated with ammonia added cautiously in very dilute solution in excess. The filtrates and washings were evaporated to about 60 c.c., and then possessed as much emanating power as 146 grms. of thoria. On evaporating the solution to dryness and removing the ammonium salts by ignition, the residue weighed 0·0583 grm. The emanating power of this residue in solution was thus about 2500 times that of ordinary thoria. In the solid state, however, the value fell to one-fiftieth. But its total radiation was equivalent to at least 23·6 grms. of thoria, *i.e.*, was about 400 times as great. It was dissolved in hydrochloric acid, and ammonia added in excess, when a precipitate weighing 0·0015 grm. was thrown down. This contained all the thorium present besides iron in appreciable quantity which had been introduced during the evaporation. It equalled in radio-activity 2·73 grms. of thoria, the ratio in this case being thus no less than 1800 times. Sodium phosphate precipitated 0·0225 grm. of white substance the activity of which was equivalent to 4·4 grms. of thoria, *i.e.*, 200 times. The sodium salts freed from ammonium still possessed a radio-activity equivalent to 3·6 grms. of thorium oxide. In other experiments, however, these had been obtained quite free from activity, and this result is due to the solubility of the phosphate in water, so that some was dissolved during the washing (which the subsequent determination of the weight rendered necessary), and appeared in the filtrate.

The radio-active residue obtained in the first place from the filtrate by evaporation and ignition, before it was re-dissolved, had, however, been tested to determine the penetrative power of the radiations emitted. If the rays from various radio-active substances are made to pass through successive layers of aluminium foil, each additional layer of foil cuts down the radiation to a fraction of its former value, and a curve can be plotted with the thickness of metal penetrated as abscissæ, and the intensity of the rays after penetration as ordinates, expressing at a glance the penetrative power of the rays being examined (compare Rutherford, *Phil. Mag.*, 1899, [5], xlvii., p. 122). The curves so obtained are quite different for different radio-active substances. The radiations from uranium, radium, thorium each give distinct and characteristic curves, whilst that of the last named again is quite different from that given by the excited radio-activity produced by the thorium emanation. The examination in this way of the penetrative power of the rays from the radio-active residue showed that the radiations emitted were in every respect identical with the ordinary thorium radiation. In another experiment, the nature of the emanation from a similar intensely active thorium-free residue was submitted to examination. The rate of decay was quite indistinguishable from that of the ordinary thorium emanation. That is, substances chemically free from thorium have been prepared possessing thorium radio-activity in an intense degree.

The main quantity of thorium hydroxide in the last experiment was re-dissolved in nitric acid, and the previous round of operations repeated twice, the filtrates from each operation being mixed and then examined exactly as in the former case. The emanating power of the concentrated solution was only equal to that of 8 grms. of thoria in this instance, and the radio-activity of the residue to that of 3 grms. From this only a small quantity of the phosphate precipitate was obtained (0·001 grm.), the radio-activity of which was equal to that of 0·3 grm. of thoria (ratio 200 : 1).

The *emanating power* of the main quantity of the hydroxide when first so prepared was 73 per cent that of thoria, *i.e.*, about one-half of its usual value. The hydroxide was converted into oxide by ignition, and its *radio-activity* compared with that of the oxide from the original nitrate prepared in the same way. It was found to be only about one-third as active, the exact ratio being 0·36 : 1.

Only one conclusion seems possible from this series of experiments. There is no longer any room for doubt that a part of the radio-active constituent ThX has been separated from thorium, and obtained in a very concentrated form, in one instance 1800 times more powerful in its actions. This result, taken into account with the reduction of the radio-activity and emanating power of the main quantity of thorium compound, and the identity of the radiations of the active thorium-free preparations with those of the ordinary thorium radiation, warrant the conclusion that ThX is a distinct substance, differing from thorium in its chemical properties, and so capable of separation therefrom. The manner in which it makes its appearance, associated with each precipitate formed in its concentrated solution, resembles the behaviour of Crookes's UrX, which he found was dragged down by precipitates when no question of insolubility is involved, and suggests the view that it is really present in minute quantity. Even in the case of the most active preparations, these probably are composed of some ThX associated with accidental admixtures probably large in proportion.

These results receive confirmation from observations made in a different method of separating ThX. The experiment was tried of washing thoria with water repeatedly, and seeing if the radio-activity was thereby affected. In this way it was found that the filtered washings on concentration deposited small amounts of material, with an activity often of the order of a thousand times greater than that of the original sample. In one experiment 290 grms.

of thoria were shaken for a long time with nine quantities, each of 2 litres, of distilled water. The first washing containing most of the sulphate already referred to was rejected, the rest concentrated to different stages, and filtered at each stage. One of the residues so obtained weighed 6·4 m.grms., and was equivalent in radio-activity to 11·3 grms. of the original thoria, and was therefore no less than 1800 times more radio-active. It was examined chemically, and gave, after conversion into sulphate, the characteristic reaction of thorium sulphate, being precipitated from its solution in cold water by warming. *No other substance than thorium could be detected by chemical analysis*, although of course the quantity was too small for a minute examination. But the absence of the substance precipitable as phosphate, noticed in the other experiments, confirms the opinion that this is an accidental admixture without influence on the qualities of radio-activity and emanating power. The penetrative power of the radiation from this substance again established its identity with the ordinary thorium radiation. In another experiment a small quantity of thoria was shaken many times with large quantities of water. In this case the radio-activity of the residue was examined and found to be about 20 per cent less radio-active than the original sample.

There remains only one step to prove beyond doubt that the radio-activity and emanating power of thorium are not specific properties of the thorium molecule—the preparation of thoria free from these properties—and on this problem we are now engaged. To sum up briefly what has already been accomplished, two different methods have effected a concentration of the activity many hundredfold in one fraction, and a corresponding diminution of activity in the remainder, but in each case the character of the radiation is not thereby affected. In one method, the active fraction appears to consist only of thorium, so far as examination has been possible, whilst in the other case radio-activity and emanating power appear to be manifested indiscriminately in all the products, without reference to their chemical nature. The simplest explanation of this behaviour on the present view, is that so far the active constituent of thorium has only been obtained in relatively minute quantity, and therefore does not answer to any definite analytical reactions.

Macdonald Physics Building,
Macdonald Chemistry and Mining Building,
McGill University, Montreal.

LONDON WATER SUPPLY.

Report on the Composition and Quality of Daily Samples of the Water Supplied to London for the Month Ending May 31st, 1902.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To Charles Perrin, Esq., M.Inst.C.E.,
Water Examiner, Metropolis Water Act, 1871.

London, June 10th, 1902.

Sir,—We submit herewith, at the request of the Directors, the results of our analyses of the 208 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from May 1st to May 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 208 samples examined by us chemically during the month, all were found to be clear, bright, and well filtered.

Although more rain has been recorded at Oxford during the past month it was below the average, and there is still a deficiency. The amount of rain that fell was 1·53 inches, the thirty-five years' average for May is 1·75 inches, making a deficit of 0·22 inch, which, added to the previous one, makes a total deficit for the year of 3·16 inches, or 36·2 per cent on the thirty-five years' average.

The various chemical analyses show that the organic matter has steadily decreased in amount since the beginning of March.

Our bacteriological examinations of 544 samples have given the results recorded in the following table; we have also examined 47 other samples, from special stand pipes, wells, &c., making a total of 591 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 26 samples) ..	193
New River, filtered (mean of 135 samples) ..	12
Thames, unfiltered (mean of 26 samples) ..	2896
Thames-derived water, from the clear-water wells of eight Thames-derived supplies (mean of 295 samples)	31
Ditto ditto .. .. highest	398
Ditto ditto .. .. lowest	0
River Lea, unfiltered (mean of 26 samples) ..	371
River Lea, from the East London Company's clear-water wells (mean of 36 samples) ..	37

A sample has been taken daily from the large filter-wells of each of the Metropolitan Companies, as has been our custom for some years. The total number of samples from all sources examined in this way during the month amounted to 446, of which 27 samples, or 6 per cent, were sterile. Twelve samples contained more than 150 microbes, while thirty samples, or 6 per cent, contained more than 100 microbes per c.c. The mean number of microbes in the thirty excess samples was 173, against a corresponding mean of 181 in thirty-one excess samples during April.

These figures show an improvement in the bacteriological quality of the London waters since April, in spite of the fact that during the last month there has been more rain in the valley of the Thames than has fallen for some time past, with the concomitant result of more surface water flowing into the river, bringing with it additional impurity.

We are, Sir,
Your obedient Servants,
William Crookes.
James Dewar.

Preparation of Anhydrous Chlorides of Samarium, Yttrium, and Ytterbium.—Camille Matignon.—The preparation of anhydrous chlorides of the rare earths in a pure state is a very delicate operation. M. Duboin for yttrium, and MM. Muthmann and Stützel for cerium, lanthanum, and the didymiums, showed that the dehydration of the hydrated salts in presence of ammonium chloride never gives a product free from oxychloride. The author now uses the method he employed to obtain anhydrous chlorides of neodymium and praseodymium (*Comptes Rendus*, cxxxiii., 289) to prepare chlorides of the other rare earths, and succeeds in preparing the anhydrous salts of samarium, ytterbium, and yttrium, the first two of these never having previously been obtained. He also finds an ytterbium chloride, $YbCl_3.6H_2O$, isomorphous with that of yttrium, and proves the existence of the three monohydrated chlorides, $SmCl_3H_2O$, YCl_3H_2O, and $YbCl_3H_2O$.—*Comptes Rendus*, cxxxiv., No. 22.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, Wednesday, May 28th, 1902.

Prof. Meldola, F.R.S., Vice-President, in the Chair.

(Concluded from p. 298).

85. "*On Phosphorus Sesquisulphide and its Behaviour with Mitscherlich's Test.*" By E. G. Clayton, F.I.C.

Many compounds of phosphorus and sulphur have been described, but of these the only one which has received any application on the large scale is the sesquisulphide, P_4S_3, discovered by Lemoine in 1864. When pure, it is a lemon-yellow crystalline solid, with a strong odour of hydrogen sulphide, soluble at 15° in 1·6 parts of carbon bisulphide, and igniting in the air at about 100°. Commercially, several qualities of this substance are manufactured. Amongst those which have come under observation have been a coarsely granular lemon-yellow powder, a fine-grained yellow powder of somewhat duller tint, and a grey product, sometimes very impure. The following are typical analyses:—

	A. Yellow (fine-grained).	B. Yellow (fine-grained).	C. Yellow (coarse grained).	D. Grey (finely granular).
Water and volatile matter (loss at 100° in an atmosphere of carbon dioxide)	0·19	0·18	8·74	7·31
Phosphorus sesquisulphide, P_4S_3	97·86	97·62	84·95	83·34
Red phosphorus	—	—	—	1·81
Phosphoric acid, H_3PO_4	1·30	1·63	2·14	1·23
Sulphur, uncombined (soluble in carbon bisulphide)	—	—	4·17	—
Sulphur, uncombined (insoluble in carbon dioxide)	0·46	0·40	—	0·17
Calcium phosphate	—	—	—	3·35
Calcium sulphate	—	—	—	0·27
Iron oxide, &c.	—	—	—	1·72
Siliceous matter	0·19	0·17	—	0·80
	100·00	100·00	100·00	100·00
Temperature at which the specimen became luminous in the dark	92°	93°	—	58°
Temperature of ignition	94°	96°	86°	72°
Reaction with Mitscherlich's test	Negative.	Negative.	Distinct.	Distinct.

Mörner has recently stated (*Svensk Farmaceutisk-Tidskrift*, 1901, xii., 177) that phosphorus sesquisulphide gives "more or less positive results" with Mitscherlich's test for phosphorus. The author has examined various specimens of commercial phosphorus sesquisulphide, and applied Mitscherlich's test to each in the following way:—Twenty grms. of the compounds were distilled with 100 c.c. of 10 per cent sulphuric acid, in an egg-shaped flask connected with a spiral condenser, the operation being conducted in a dark room. The very small amount of light emitted by the lamp was screened from the condenser and receiver, which were in complete darkness. In each case, 40 c.c. of liquid were distilled over. The results with comparatively pure specimens, such as A and B, were absolutely negative, not the faintest luminosity being perceptible in any part of the apparatus. It is evident that pure, or even approximately pure, phosphorus sesquisulphide gives no reaction with Mitscherlich's test, and that Mörner's results were obtained with samples containing small quantities of phosphorus, or of phosphorous oxide. Very crude specimens of phosphorus sesquisulphide no doubt occasionally give Mitscherlich's reaction; thus, with the impure specimen, D (tested after being kept for many months in the laboratory), a distinct luminous cloud played about the exit of the condenser, and in the air of the receiver above the surface of the distillate, which had a faint odour of phosphorous oxide. The sample C, which had been kept for nearly three years, and had undergone considerable oxidation, being quite moist, also gave a decided reaction. The distillate from this also smelt distinctly of phosphorous oxide. The specimens A and B, which gave negative results, had been recently made. Even in these samples some oxidation had occurred, but apparently no phosphorous oxide was present. The author is now conducting some experiments with the object of discovering whether exposure and keeping can so induce partial oxidation in, or alter the composition of, pure, or nearly pure, phosphorus sesquisulphide as to impart to it after a time the property of giving Mitscherlich's reaction. Apart from its scientific interest, the point was of considerable practical importance. Meanwhile, the absence both of free phosphorus and of phosphorous oxide from phosphorus sesquisulphide of fair quality and purity and comparatively recent manufacture was clearly indicated by the negative reaction with Mitscherlich's test, and the following circumstances were corroborative as far as they went. Phosphorus sesquisulphide may be subjected to friction in air at a temperature very considerably exceeding the ignition-point of phosphorus without becoming luminous, and with a total absence of "phosphorus fume"; it neither ignites nor begins to glow in the dark until a temperature of 92—96° is reached, and the finely-divided residue of a solution in carbon bisulphide, evaporated on filter-paper at the ordinary temperature, neither glows in the dark, fumes, nor ignites spontaneously.

86. "*Atomic and Molecular Heats of Fusion.*" By P. W. Robertson.

No satisfactory relationship has hitherto been found connecting the latent heat of fusion of substances with their atomic or molecular weights. In the case of the elements, the following is shown to yield satisfactory results:—"For the elements with atomic weights above 40 which do not expand on freezing, the atomic heat of fusion divided by the melting-point on the absolute scale into the cube-root of the atomic volume is a constant." That is,—

$$Aw/T\sqrt[3]{V} = \text{constant.}$$

The numbers have a mean variation of ± 10 per cent. The only exception is lead, for which the value of the expression is 25 per cent below the mean.

In the case of the binary inorganic compounds also, the expression—

$$Mw/T\sqrt[3]{V},$$

where V is the specific volume of the solid, yields concordant results. The values of the expression for organic compounds increase with the number of atoms in the molecule, but when compounds of similar constitution are considered, fairly constant results are obtained.

To test these relations, the latent heats of the following substances were determined:—

Thallium	= 7·2	Phenanthrene	= 25
Lead	= 6·45	Phenylacetic acid	= 32
Tin	= 14·05	Tribromophenol	= 13·4
Dinitrobenzene	= 29·0	Tribromoaniline	= 14·4
		Thiosinamine	= 33·4.

87. "*The Preparation of Mixed Ketones by Heating the Mixed Calcium Salts of Organic Acids.*" By E. B. Ludlam.

A device for preparing a simple ketone from the calcium salt of the corresponding organic acid was described by Young (*Trans.*, 1891, lix., 623), which consisted in decomposing the salt at the temperature of boiling sulphur and removing the products by a stream of carbon dioxide.

The author has extended the method to mixtures of calcium salts with very satisfactory results.

The conclusions arrived at are—(1) That the decomposition is not of one molecule, but of two; (2) there is a greater tendency towards the production of the mixed than of the simple ketone; (3) that, for economy, it is advisable to distil the more expensive salt with excess of the cheaper one. By so doing the yield of mixed ketone from a given weight of the expensive salt is increased.

88. "*Isomeric Additive Products of Methyl, Ethyl, and Propyl Benzyl Ketone with Benzylidene Aniline.*" Part IV. By F. E. FRANCIS and E. B. LUDLAM.

The benzylideneaniline additive products of methyl, ethyl, and propyl benzyl ketones were prepared in the expectation that they would furnish evidence as to the constitution of the tautomeric forms which such compounds are capable of assuming. Methyl benzyl ketone contains the group $Ch_3{\cdot}CO{\cdot}CH_2$—, which in ethyl acetoacetate gives rise to the tautomeric form $CH_3{\cdot}C(OH){:}CH$—, and it was hoped that the work would have contributed to the solution of the problem of the constitution of the tautomeric forms of that compound.

Methyl benzyl ketone gave with benzylideneaniline an additive product melting constantly at 173° (*a*). This, under the influence of piperidine, gave the *β*-modification melting at 182°, and another modification melting at 184° when subjected to the action of a trace of sodium ethoxide. The hydrochloride of the *α*-modification was unstable; it gave no fixed melting-point, but slowly decomposed between 140° and 190°. Propyl benzyl ketone gave a similar *α*-product, crystallising in silky needles melting at 136°, rising to 142° under the influence of piperidine, and to 143° with sodium ethoxide. Ethyl benzyl ketone gave an *α*-product melting at 161°, intermediate between 136° and 173°. Neither the original ketones nor any of the additive products gave any colouration with ferric chloride, and the melting-points of the *β*- and *γ*-varieties fell when they were re-crystallised from pure benzene. They were thus too unstable to serve the purpose for which they were prepared.

89. "*The Influence of Solvents on the Rotation of Optically Active Compounds.* Part III. *Influence of Benzene, Toluene, o-Xylene, m-Xylene, p-Xylene, and Mesitylene on the Rotation of Ethyl Tartrate.*" By T. S. PATTERSON.

It was found that in dilute solution the influence of benzene is to lower slightly the specific rotation of the dissolved ethyl tartrate. Toluene has a greater influence of the same kind, and this influence becomes greater and greater in the other solvents in the order in which they are placed above.

It was found also that in benzene, toluene, *o*-xylene, and *m*-xylene there exists, not only a concentration of minimum rotation, but also one of maximum rotation, which lies at a greater dilution. The concentration-rotation curves for *p*-xylene and mesitylene show only a point of inflection.

The relationship between molecular-solution-volume and rotation has been investigated, and it was found that in general, although not without exception, the behaviour was the same as in the monohydric alcohols; when the dissolved ethyl tartrate has a molecular-solution-volume greater than the molecular volume of the pure ester the rotation is correspondingly low.

The relationships between rotation, latent heat of vaporisation of the solvent, and surface-tension of the solvent were also discussed.

90. "*The Influence of Solvents on the Rotation of Optically Active Compounds.* Part IV. *Influence of Naphthalene on the Rotation of Ethyl Tartrate.*" By T. S. PATTERSON.

Naphthalene was found to differ greatly in its behaviour from the benzene hydrocarbons. It has the effect of greatly increasing the rotation of the dissolved ethyl tartrate, and its influence is similar to that of water in this respect as well as in the effect of temperature change.

CORRESPONDENCE.

VALENCY AND RADIO-ACTIVITY.

To the Editor of the Chemical News.

SIR,—I trust you will permit me to publish the skeleton of a theory connecting valency with the property of radio-activity, which I have been elaborating since the end of 1900. The first communication I made to your columns on this matter was a letter which appeared in the CHEMICAL NEWS of March 15th, 1901 (vol. lxxxiii., p. 130). In that letter the term "ion" should be replaced by "electron."

So many authors have lately been approximating to these views that I feel it would be dangerous to delay publishing this preliminary note until I have completed the experiments to confirm or disprove it that I am carrying out in the physical laboratory here—experiments which, from the nature of the case, must occupy a considerable time.

The degree of valency that an elementary atom manifests is conditioned by the number of electrons it contains, according to the universally accepted theory of Helmholtz. For instance, an atom of iodine is tri-valent when there are condensed on its surface three negative electrons, while it is mono-valent when it holds but one. So that a change from a tri-valent to a mono-valent condition must be attended with the loss of two negative electrons.

But a consideration here forces itself on our attention. Electrons are of two kinds—positive and negative. The same atom contains both kinds, and in general different numbers of each. This is shown by the fact that one and the same atom acts with a quite different degree of valence towards positive and negative radicals, as Mendeleeff long ago pointed out. Further, quite a definite law is followed in this matter. If n represent the number of positive electrons and n' the number of negative, Mendeleeff found in any one case that $n+n'=8$.

These positive and negative electrons do not, in the same atom, combine with each other and neutralise one another's effect, but each set apparently retains its position on the atom as if the other set were absent, so far as can be judged from the phenomena of chemical combination.

Again, the same atom has in general a different attractive power for the two different kinds of electrons, some atoms attracting positive electrons with a force greater than that which they attract negative, and *vice versâ*. To this cause Helmholtz referred the difference of potentials developed when two different metals are placed in contact.

Metals hold negative electrons with a far greater degree of feebleness than non-metals.

If, therefore, radio-activity is produced by the giving off of electrons this emission could be produced from them in two ways:—

1. By heating up the element. This by increasing the kinetic energy of the electrons would cause them ultimately to be cast forth by the atom. I have considered this point in a previous communication.
2. By a change of valency of the element in combination.

It is the last point I wish to consider here.

In general, when a metallic atom enters into combination with another more negative atom, it is the positive electrons on the first atom that are in the main responsible for the union; *e.g.*, the combination, $FeCl_3$, may be represented thus, $\overset{+++}{Fe}\ \overset{---}{Cl_3}$. It might be concluded, therefore, that if by some means we reduced the valency of the iron atom from $\overset{+++}{Fe}$ to $\overset{++}{Fe}$, keeping the valency of the Cl constant, we should get liberated a positive electron thus:

$$\overset{+++}{Fe}\ \overset{---}{Cl_3} = \overset{++}{Fe}\ \overset{--}{Cl_2} + \overset{-}{Cl} + (+).$$

But the case is far more complicated than this. (*a*) In the first place Professor J. J. Thomson has shown that

the mass of the positive electron is great compared to that of a negative electron, and it may be doubted whether free positive electrons are ever thrown off from the atom, or could be, since there is reason to believe that what is known as "positive" electricity is a fundamental property of the matter forming the atom, in the same way that gravity is.

So that when $\overset{+++}{Fe}$ is reduced to $\overset{++}{Fe}$, as in the preceding compounds, it may be justly doubted whether there is a loss of electrons. It seems much more probable that there is an addition of negative electrons to the iron atom over and above that which it normally holds in the ferric condition, and which has the effect of masking the action of a positive valency bond.

So that the operation of reducing $\overset{+++}{Fe}$ to $\overset{++}{Fe}$ is not to be represented thus:—$\overset{+++}{Fe} = \overset{++}{Fe} + (+)$; but rather thus:—$\overset{+++}{Fe} + (-) = \overset{+++-}{Fe}$.

A reduced atom is therefore often to be regarded as suffering, not from a deficiency of electrons, but rather from an excess of electrons of an opposite polarity. Since at normal temperatures each atom can retain eight electrons, irrespective of sign, any number above this would be held with great feebleness.

So that on heating up the reduced Fe atom we should get off the added negative electrons. In support of this view I urge the following facts:—

1. When cathode rays fall on a chemical compound they reduce the central element to a lower degree of valence.
2. When compounds thus reduced are heated up to a temperature of 100° or more they throw off again the negative electrons thus supplied (*vide* McLennan, *Phil. Mag.*, 1902, [6], iii., 195—203).
3. Russell has shown that unsaturated carbon compounds often throw off an emanation that affects a photographic plate in the same way that radio-active bodies do (*vide* Chemical News, 1897, lxxv., 302; 1898, lxxvii., 163; 1899, lxxix., 121, 133; lxxx., 288.

I hope to begin experiments on this subject shortly.

(*b*) In the second place, there is grave reason to believe that when one atom combines with another, both the positive and negative electrons contained on each atom come into play. For instance, when an atom, A, combines with another decidedly more negative atom, B, it acts with one degree of valence. And when it combines with a decidedly more positive atom, C, it acts with a totally different degree of valency. In the first case the positive electrons of A are mainly instrumental in deciding the union; in the second case its negative electrons.

If now this atom, A, enter into combination with another atom, D, which is intermediate in electrical polarity between the extreme positive and negative atoms B and C, A will act with a valence intermediate between the valencies which it exhibits towards B and C.

It seems, therefore, in this case, that both the positive and negative electrons contained in A come into action with the negative and positive electrons contained in D. This is probably quite a general phenomena.

A chemical combination therefore is no simple affair. It is a complicated dual reaction between the positive and negative electrons contained on each atom—the complication being increased by the fact that the electrons of opposite polarity are retained on one and the same atom with very different degrees of stability, so that we may get a surging of negative electrons to one atom, and, consequently, the appearance of positive electrons on the other atom, in a manner that makes it difficult to say what would precisely happen were the valency of the elements forming the compounds reduced.

One fact, however, stands out clearly.

Metals hold electrons, negative electrons at least, with a very much greater degree of instability than non-metals, as is seen, not only in their power to conduct electricity, but also in the unstableness of their compounds, as is seen in the readiness with which they dissociate in solution; the stability of the union being in the main decided by the stability with which the negative electrons are retained by the metallic atom.

Therefore, if radio-activity is conditioned by the emission of negative particles, a gentle warming of a compound formed by the union of one metal with another should lead to the evolution of such particles from the first metallic atom, as the compound progressively dissociates with the rising temperature, and therefore as the first metallic atom becomes reduced to a lower degree of valence.

1. The fact that Hg when contaminated by a trace of Na or Zn becomes capable of acting readily on a photographic plate in the dark confirms this view. I am carrying out experiments on this subject in the physical laboratory here.
2. When H_2O_2 decomposes into H_2O and O, the hydrogen atom is probably changing its valence from 2 to 1, and therefore may be expected to evolve electrons. I am at present engaged in looking for this effect. The fact that (*a*) it gives off an emanation which acts on a photographic plate in the same way that radio-active bodies do (*vide* Russell; I have repeated and confirmed his observations); (*b*) and that it rapidly loses a charge of negative electricity from its surface when undergoing decomposition (*vide* D'Arcy, *Phil. Mag.*, 1902, [6], iii., 42—45) makes this view extremely probable.

The preceding is a mere outline of the general theory. I may say I would not have ventured on this note until I had completely proved or disproved it by experiment were it not that several papers have recently appeared in which there is a distinct approximation to such ideas—I am, &c.,

Geoffrey Martin.

University of Berlin, June 10, 1902.

NOTICES OF BOOKS.

The Hydro-metallurgy of Copper; Being an Account of Processes Adopted in the Hydro-metallurgical Treatment of Cupriferous Ores, Including the Manufacture of Copper Vitriol; with Chapters on the Sources of Supply of Copper and the Roasting of Copper Ores. By M. Eissler, M.E., Member of the Institute of Mining and Metallurgy. London: Crosby Lockwood and Son. 1902. Pp. 228.

The hydro-metallurgical treatment of copper ores was originated at the Rio Tinto mines several centuries ago. Several processes have since been evolved, principally through the investigations of manufacturers of sulphuric acid, who made it a special study to extract the low residual percentage of copper from the burnt pyrites and cinders. The application of the various lixiviation processes resulting from the successful solution of the problem of leaching low-grade copper ores are likely to have more attention given to them now, owing to the growing necessity of working the low-grade copper deposits in various parts of the world.

The author makes no claim to originality in the present work, but he has collated and described the various processes now known to be in operation in an admirable manner, though detailed information has often been denied to him for obvious commercial reasons.

The book is divided into three parts and seventeen chapters. Part I. is on the production of copper and its sources of supply, and includes not only wet but dry smelting processes. Part II., which forms the main feature of the work, is on methods of hydro-metallurgical treatment of copper. Under favourable circumstances ores which contain only ½ or 1 per cent of copper are treated by these methods, especially when their treat-

ment is in connection with some other industries, such as the manufacture of sulphuric acid. Not every low-grade copper ore is found to be adapted to hydro-metallurgical treatment, owing to impurities which interfere with the chemical reactions, those most suitable being poor oxides and sulphides in a quartz gangue. Rich by-products, matte, black copper, &c., are treated by the wet process, as well as ore heaps which have undergone decomposition by atmospheric agencies, and mine waters from copper-mines.

The wet processes for the treatment of copper ores are employed for:—(1) Ores which contain the copper as oxides or carbonates; (2) those which contain the copper as a sulphate; and (3) those which contain the copper as a sulphide. The oxides of the first class are usually found associated with carbonates. Soluble sulphates also are rarely found in large quantities, but are formed as mentioned above by atmospheric influences. The third class, the sulphides, are roasted and rapidly converted into a soluble condition.

The author then goes on to explain the various methods adopted for special ores, and the means of recovering the copper from solution. Part III. is on the roasting of copper ores, and includes a large number of diagrams of furnaces and appliances in use for this purpose.

The book is well written, and will undoubtedly be of interest and use to those engaged in the copper industry, especially to students, who will find a large amount of information given in a clear and straightforward style.

There is an index, though not a very extensive one.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxiv., No. 22, June 2, 1902.

Electro-capillary Properties of Organic Bases and their Salts.—M. Gouy.—In a previous research the author showed that salts of the same acid give in equivalent solutions electro-capillary curves differing very slightly from one another whatever the base of the salts used. He now extends this investigation to salts of organic bases and also sulphates, phosphates, and bromides. The effect produced can be analysed more in detail by operating on mixtures. For example, a normal solution, L_1, of H_2SO_4 is taken and its electro-capillary curve traced. The greatest curve is obtained with Hg_2SO_4 in a normal solution of Na_2SO_4, which remains unchanged. To L_1 a small quantity of a base is added; for example, amylamine. We have then the case of a solution L_2 containing a little amylamine sulphate with a large excess of H_2SO_4. The electro-capillary curve of L_2 may be confused with that of L_1 for the positive branch, but separates on approaching the maximum, and remains below for the negative branch.

Ammoniacal Cupric Oxide.—M. Bouzat.—The author determines the heat of formation of ammoniacal cupric oxide and its heat of neutralisation by various acids, and he finds (1) that the ammoniacal cupric base is formed from cupric hydrate and ammonia, with a slight evolution of heat; (2) that this base is a very strong one, much stronger than ammonia, this latter fact being verified by the phenomena of displacement in which it takes part.

Action of Monochlorated Ethyl Acetylacetate on the Diazoic Chlorides.—G. Favrel.—Monochlorated ethyl acetylacetate reacts on the diazoic chlorides in the same way as ethyl methylacetylacetate and ethyl ethylacetylacetate. Elimination of the acetyl group takes place, and formation of hydrazones, in the composition of which the substituted radical (which is in this case chlorine) is joined to the hydrogen atom of the acetylacetic ether. Using the same method it is found that bodies having the composition of hydrazones derived from ethyl chloroxalate can be obtained. The author proposes to continue his research on the properties of this body.

Certain Salts of Benzylamine.—René Dhommée.—The author prepares a number of salts of benzylamine, the chief being—Benzylamine nitrate, $C_6H_5—CH_2NH_2,NO_3H$; the acid sulphate, $C_6H_5—CH_2NH_2,SO_4H_2$; the borate, $(C_6H_5—CH_2NH_2,2B_2O_3)_2+3H_2O$; the neutral chromate, $(C_6H_5—CH_2NH_2)_2CrO_3+2H_2O$; the neutral oxalate, $(C_6H_5—CH_2NH_2)_2C_2O_4H_2$; the benzoate,—

$$C_6H_5—CH_2NH_2,C_6H_5—CO_2H.$$

MISCELLANEOUS.

Chemical Handicraft.—We have received from Messrs. John J. Griffin and Sons a copy of their new 1902 Catalogue. The firm has spared no pains or expense to make the book as complete as possible; it is profusely illustrated, and includes many new pieces of apparatus.

The Solubility of Salts; Telluric and Allotelluric Acids.—J. Mylius.—The author compares first the hydrates of tellurate of sodium, $TeO_4Na_2+2H_2O$ and $4H_2O$, with the corresponding molybdates, chromates, &c.; the first occurs in hexagonal plates soluble in 130 parts of water at 18°, and in 50 parts of water at 100°; the solutions have a very alkaline reaction, and are precipitated by carbonic acid; the second hydrate is less stable, and is soluble at 18° in 70 parts of water, and at 50° in 40 parts. There also exists a basic tellurate, $TeO_5Na_4+8H_2O$. Felted needles form in the presence of an excess of concentrated soda; they are hydrolysed by pure water regenerating the neutral salt. There is also to be obtained a basic lithium salt, $TeO_5Li_4+4H_2O$, but no defined neutral one. On examining the solubility of free telluric acid the author gives the curves relating to the different hydrates, $TeO_4H_2+6H_2O$ and $2H_2O$, which cross at about +10°; he then considers the total or partial dehydration of these hydrates by heat. The most stable hydrate, $TeO_4H_2+2H_2O$ or $Te(OH)_6$, calcined at 350°, gives, as is well known, the insoluble yellow anhydride, TeO_3; but if heated only to 140° it undergoes aqueous fusion, giving a syrup in which a white, amorphous, granular material is gradually deposited, which only re-dissolves in water with difficulty, and not then regenerating ordinary telluric acid. The solutions of this product differs again from those of TeO_4H_2 by their more distinctly acid taste, their precipitation by alkalis and by carbonate of guanidine, and their electric conductivity. The author admits the existence of a polymer $(TeO_4H_2)_n$, comparable to metaphosphoric acid, and gives it the name *allotelluric acid*. Finally, he observes that persons who have absorbed a small quantity of tellurium (even 1 m.grm. only) are troubled with a metallic taste which persists in the mouth for several days, and at the same time their breath acquires a penetrating odour of garlic; these facts are probably due to the formation of telluride of methyl in the organism.—*Berichte*, vol. xxxiv., p. 2008.

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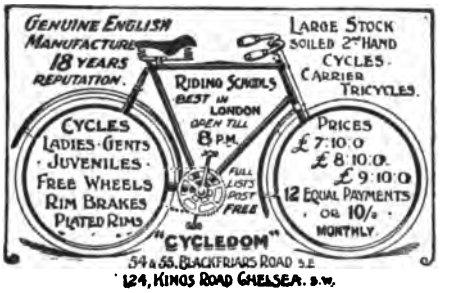
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